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TECHNICAL MEMORANDUM

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TITLE: Operation and Post-Test Inspection of the SNAP-8 Pre-Prototype Boiler P/N CF 751840, S/N 2

ABSTRACT

The subject boiler, designed and built by NASA-LeRC was tested in the AGC PCS-1 facility for 8700 hours and 27 startups. During this period, a bellows used for stainless steel-to-tantalum differential expansion failed. The primary NaK loop shell failed twice and a static NaK stainless steel tube cracked in the same area as the primary NaK shell failures. Diminution of mercury side pressure drop during operation was attributed to mercury loop contamination, not boiler design.

Tantalum used for mercury containment was shown to be an ideal material metallurgically.

With some design modifications, it appears that this boiler could meet the AGC Specification 10621 requirement of 10,000 hours of operation with 100 startups.

KEY WORDS: tantalum boiler, transition joint, double-containment, bare-refractory

APPROVED:

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E. S. Chalpin

NOTE: The information in this document is subject to revision as analysis progresses and additional data are acquired.

AEROJET-GENERAL CORPORATION

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I. INTRODUCTION

The BRDC (Bare-refractory, double-containment) Boiler P/N CF751840 is a recent design of NaK-to-Hg heat exchangers developed for use in the Rankine cycle power conversion system. The attainment of adequate heat transfer performance and material compatibility with the working fluids, particularly in the boiling mercury region, have been two major problems encountered during previous development of boilers.

The first boiler built and tested was a single-pass tube-in-shell design. Four tubes were helically coiled and contained in a cylindrical, toroidal shell. Heat transfer performance was not attainable, due in part because the NaK flow was in a combination cross-flow and counter-flow pattern relative to the mercury flow.

The second generation boiler fabricated was a single-pass, tube-in-tube boiler with pure counter-flow. Seven mercury containment tubes were placed within a larger NaK containment tube with the assembly coiled in a helical pattern. Each of the seven small tubes contained an inlet plug insert and swirl wire. This design exhibited better heat transfer and dynamic performance.

The materials used in these boilers were 9% Cr - 1% Mo steel for the mercury containment tubes and Type 316 or Type 321 stainless steel for NaK containment. Results of small scale tests indicated 9% Cr - 1% Mo steel had good resistance to Hg corrosion. However, upon disassembly of these boilers after operation between 1000 to 2000 hours, it was found that the boiler tubes showed excessive Hg-side corrosion and NaK-side embrittlement. The NaK containment material was observed to be in good condition. In addition, the boiler performance data indicated at certain times, usually during system start-up, that the mercury was not wetting the tube wall. This non-wetting in the boiler results in high liquid carry-over, reduced boiler pressure drop, reduced superheat and a longer required boiling length. Investigation of this phenomenon showed the degree of wetting (referred to as boiler conditioning) depends on the type of containment material, the cleanliness of the surfaces and the amount of contamination carried by the mercury to the boiler during system operation.

From further metallurgical tests conducted on various materials, tantalum was chosen as the mercury containment material because of its low solubility, high corrosion resistance and good wetting properties in mercury. Concomitant investigations were carried on with the SB-1 test boiler, which was an experimental Ta/316SS, double-containment, 1/7 scale unit (single tube). The test results indicated that tantalum experienced no mercury corrosion, erosion, or other deleterious effects when subjected to the required SNAP-8 operating conditions. It was also established that two important requirements for proper boiler conditioning were cleanliness of the boiler tubes and the need for a vacuum tight system to prevent surface oxidation and contamination.

The favorable results of the static and dynamic tests with tantalum prompted the design and fabrication of a bi-metallic tube-in-tube boiler at AGC. This boiler was similar to the 9 Cr - 1 Mo tube-in-tube boiler in length, number of tubes, etc.,; however, the mercury containment tubing utilized a stainless steel tube implosively bonded to a tantalum liner. The stainless steel tube provided the tantalum liner with structural strength plus protection from erosion by the flowing NaK. Three bi-metal boilers were designed; however, only one was fabricated.

During the design period of these bi-metal boilers, NASA-LeRC designed and fabricated a counter-flow, tube-in-tube boiler similar in geometry and heat transfer concepts to the previous tube-in-tube designs. The design featured an all-tantalum tube and header configuration for the mercury with a static NaK layer surrounding each tantalum tube. This static NaK layer was contained by a stainless steel oval tube and header arrangement. Termed "double-containment", this concept serves to meet the requirements of a man-rated system which specifies double isolation between the radioactive primary NaK and the mercury loop.

Three boilers of this type were built by NASA-LeRC. The first has been tested at the General Electric Company for 15,125 hours and 6 starts, the second was tested at AGC for 8,700 hours and 27 starts, and the third at NASA-LeRC for 157 hours and 135 starts. The remainder of this memorandum will deal with the #2 boiler's long term performance, test events and post-test investigation with results and conclusions thereof.

II. SUMMARY

The boiler exhibited good mercury side outlet pressure stability during operation in PCS-1; however, the mercury pressure drop declined from 200 psid to 130 psid over the total operating time of 8,700 hours.

Buildup #1, of Boiler #2, experienced primary NaK shell and bellows failures which necessitated a redesign of both the Hg inlet and outlet. Buildup #2 had two more failures, namely, another primary NaK shell failure and a cracked 321SS static NaK tube. The shell failure was repaired but the tube failure was not because it was not detrimental to safe boiler operation.

Following the 8,700 hour test, the plan to repair the 321SS tube failure was found to be unfeasible when the severity of the failed tube was appraised and a piece of the boiler's 90% Ta - 10% W swirl wire was found in the turbine alternator assembly inlet filter downstream of the boiler. The mercury inlet section including ten inches of the tube bundle was removed from the boiler for analysis and study.

The results of investigations led to the following conclusions:

1. Tantalum is superior to 9% Cr - 1% Mo steel as the containment material. No cracking, erosion or corrosion was found in the tantalum areas examined.

2. The principle of double-containment was proven in this design, irrespective of the eventual failure of the static NaK tube. A single failure between the primary NaK and Hg automatically terminated testing with earlier designs.

In order to meet the 10,000 hour life and 100 start requirement, the following problems would have to be solved:

1. Debonding of the Hg inlet Ta/316SS co-extruded tapered transition joint.

2. Hydrogen embrittlement of the 90 Ta - 10W swirl wire.

3. Excessive thermal gradients and stresses between the NaK outlet port and the 316SS header.

4. Sources of mercury loop contamination would have to be traced and eliminated or minimized.

III. BOILER OPERATION

A. Performance

During the entire test period, Boiler #2 performance data indicated good conditioned operation with regard to liquid carry-over, terminal temperature difference and boiler stability. The liquid carry-over was 5%, the terminal temperature difference was 30°F and the outlet pressure oscillations were ± 1% at a design mercury vapor flow of 12,300 lb/hr. The total mercury side pressure drop was an unpredictable performance parameter, however. Figure 1 graphically presents variations in pressure drop with changes in mercury flow at a given NaK flow and NaK inlet temperature. Curves numbered (1) through (4) are data taken during the boiler operation between initial startup and the time of the boiler differential expansion bellows and primary NaK shell failure (1313 hours). The curves numbered (5) and (6) represent pressure drop data over the remaining 7,387 hours of operation. The significant differences between curve (1) and curves (2) through (6) are the pressure drop relationship with mercury flow. Curve (1) is indicative of boiler performance with effective plug inserts where most of the boiler pressure drop occurs. As the mercury flow is increased, a maximum is reached whereby any further increase in mercury flow will reduce plug insert exit quality. Hence, more boiling is forced to take place in the unplugged tube region (with a much larger cross-sectional area) thus reducing the boiler pressure drop. When the plug insert becomes less effective, i.e., reduced heat transfer, increased mercury flow results in a pressure drop versus flow slope that remains positive as shown by curves (2) through (6). The differences in magnitudes of the pressure drop at each given flow are a function of how much heat transfer degradation in the plug insert has taken place. The fact that the unplugged tube length was over-designed allowed the boiler to behave in a stable manner and still produce the required superheat and vapor quality. After a major repair and boiler cleaning at 1313 hours, the boiler pressure drop returned to a value of 175 psid as shown on curve (5). This value was maintained for 6,000 hours of continuous testing until a primary NaK-to-air leak in the boiler forced a shutdown. Following the repair, the pressure drop returned to only 140 psid at design flow, evidence that further heat transfer degradation had taken place.

A representation of the effectiveness of the plug inserts can be made by means of the NaK temperature variation along the boiler as shown in Figure 2. The three profiles shown correspond to the time periods from which data were taken for curves (1), (5), and (6) of Figure 1. From these profiles, it can be seen that a significant difference exists between the three cases. The major differences exist in the slope of the curves in the plug insert region and the available superheat length. The flat portion of the curves in the plug insert region are indicative of little heat transfer which forces most or all of the boiling to take place in the unplugged tube section, thus reducing the superheat length. The degradation of plug insert effectiveness, i.e., heat transfer, could be caused from enlargement of the plug flow passages by mercury corrosion, erosion, by a film of surface oxides and/or contaminants or by a combination of all three. Post-test investigation of the Hg inlet tantalum surfaces revealed no corrosion or erosion had taken place. BRDC Boiler #1 at the G. E. Co. test facility has operated in excess of 15,000 hours with no pressure drop or heat transfer degradation. Since this facility does not contain sources of oil contaminants, it was not expected to degrade. BRDC #3 at the LeRC W-1 facility, which is similar to PCS-1, has operated for many more startup/shutdown cycles than was experienced by Boiler #2. These tests are being examined in greater detail to determine if pressure drop decay had occurred. Examination of BRDC #1 pressure drop versus mercury liquid flow relationship shows it to behave in the same manner as curve (1) of Figure 1 for BRDC Boiler #2. Likewise, the G. E. Co. also reported mercury outlet pressure oscillations within $\pm$ 1% and terminal temperature differences of 10° to 20°F.

Two performance mappings were made. Unfortunately, they were not made when the boiler was operating at the peak of its conditioned performance. The data was taken during boiler stability tests (April 17 and 18, 1969) and reactor deadband tests (August 22, 28, and 29, 1969). The data presented in Figures 3 to 6 are from the April 17 and 18 tests since the boiler performance was not degraded to the extent of the later boiler mapping tests.

B. Test History

Figure 7 is a time plot of the operational history of BRDC Boiler #2 in PCS-1 from initial installation in March 1968 to the planned shutdown on 22 September 1969. The time plot includes all events encountered during operation with the cumulative operating time that had elapsed between these significant events. The change in build-up numbers from #1 to #2 resulted from extensive repairs due to failure of the bellows and the NaK-to-air leakage at the NaK outlet port and shell which occurred after 1313 hours of testing. After the repairs were made, the boiler was chemically cleaned according to the procedure given in AGC Spec. 1031918. This was the only time the boiler was cleaned, other than the initial cleaning following fabrication. Figures 8 and 9 show the configuration of both ends of the boiler for buildups #1 and #2, respectively.

The primary NaK-to-static NaK leak was suspected when the test engineer noted a 10 psi rise in the static NaK expansion reservoir at 2748 hours of operation. Confirmation of the leak was made by observing that a change in primary NaK loop pressure was accompanied by a corresponding change in the static NaK pressure. Reference (2) stated that further tests were conducted to determine the size of the leak and that from this test data the leak has an equivalent diameter of 0.17 inch. After another month of testing, Reference (3) stated no change had occurred in the size of the leak. No repair of this leak was deemed necessary and the boiler continued to operate satisfactorily.

A NaK-to-air leak occurred through the type 316SS shell adjacent to the NaK outlet port after 4638 hours. This failure (F.R. #1195) was repaired in place with a "clam-shell" patch made of type 316SS material. The patch was welded to the boiler over the failed area as shown in Figure 10 and no additional problems were experienced during the remainder of the test period.

IV. POST-TEST INSPECTION

Following the 22 September 1969 shutdown, the plan was to repair the static NaK-to-primary NaK leak and measure the tantalum parts that were exposed during the repair. However, the discovery of a piece of the 90 Ta - 10 W boiler swirl wire found in the TAA inlet filter prompted a more extensive investigation of the boiler.

A. Boiler Disassembly

Pictorial evidence and physical dimensions as to the condition of the boiler can be seen in Figures 11 through 25. Comments and observations noted for each figure follow:

Figure 11 - The encrustation of the bottom static NaK tube as viewed through the primary NaK outlet was found to be NaK oxides with very little mass transfer deposit. This picture was taken after removal of the boiler from PCS-1 and prior to cleaning and disassembly.

Figure 12 - The mercury inlet bellows, the zirconium corrugated foil and the tantalum dome can be seen in this photo. Some squirming can be seen in the bellows but this was present when they were originally installed. They are shown in the collapsed position because that is their attitude when the boiler is cold. When the boiler heats up, the bellows extend approximately one-half inch, as measured during test. The zirconium foil was badly decomposed and fragmented. The analysis of the foil for oxygen, hydrogen, and carbon is shown in Appendix B. The tantalum dome appeared to be in excellent condition.

Figure 13 - The zirconium foil was removed from the mercury inlet to examine the tandem tantalum-to-316SS transition joint. This joint was ultrasonically inspected for debonding. The black wavy circumferential line shows the extent of the debonding which encompassed approximately one-half the circumference. The highlighted wavy circumferential line to the left of the black line is the runout of the stainless steel over the tantalum. It was estimated that approximately one-half of the joint is debonded at its worst. Dye-check of the area and the dome showed no cracking of the dome or welds but some indications were noted at the tantalum-to-stainless steel interface. Dye-check revealed no cracking in the bellows.

Figure 14 - The mercury outlet tantalum tubes, tantalum dome, zirconium corrugated foil and stainless steel header are shown. The indentation that can be seen on the tantalum dome was present when the boiler was first assembled. The zirconium foil is in a much better condition here than that shown at the mercury inlet.

Figure 15 - The zirconium foil was removed from the mercury outlet to examine the brazed tantalum-to-stainless steel transition joint. Dye-check of all parts indicated no cracking in any of the parts.

Figure 16 - This photo shows the 321SS static NaK tube bundle, the 316SS header, the two baffles and the tube bundle support. The center static NaK tube, as shown, cracked just to the left of the right baffle.

Figure 17 - This photo shows the cracks in the static NaK tube with the baffle moved to the left. This section of the tube was removed for metallurgical analysis by the Materials Group (see Appendix B). The stippled area to the right of the cracks was found to be mass transfer deposits. When the cracked section of the tube was removed it was noted that the tantalum tube underneath (shown in Figure 18) was eroded or "washed out" to a depth of .010-.020". This anomaly occurred during boiler operation at temperature, as borne out by a dimensional stackup of the parts. In the cold condition, the tantalum tubing eroded area was 0.537" to the left of the crack as shown in this photo.

Figure 19 - The crack is shown looking at the boiler static NaK tube 90° from that shown in Figure 17. The circumferential crack had propagated approximately 3/4 of the way around the tube and was in the area where the tube commenced to go from circular (to the left) to elliptical. The adjacent surfaces of this crack appeared to be washed out in a similar manner to that noted on the tantalum tube underneath. The other 6 static NaK tubes showed no cracks or distress when examined with dye-check.

Figures 20 and 21 - They show the dimensional checks of the boiler inlet and outlet tantalum parts, respectively. It can be seen that no discernible creep is in evidence after 8700 hours of test. The dimensions shown were compared to the drawing only, since actual measurements were not made at the time of assembly.

The finding of the piece of boiler swirl wire in the TAA inlet filter prompted the inspection of the mercury outlet tubes with a Flexiscope (flexible boroscope). Because of geometry and the limited flexibility of the flexiscope, only the center tube could be inspected. The swirl wire was found to be in tact from the end of the tube to a point at which the primary NaK inlet port attached to the boiler. At that point, a portion of the wire was missing. The Materials Group has analyzed the piece of wire and compared it to new wire. They have also performed tests with new wire and the cleaning process noted at 1313 hours on Figure 7. Their findings are given in Appendix A.

The mercury inlet tantalum dome was cut on a circumference just upstream of the dome-to-header weld. Visual inspection revealed no contamination in the dome, the header or the orifices. It was noted that the protrusion of each orifice varied, one to the other relative to the header plate. This was done during fabrication. The orifice-to-tube-to-header welds indicated that large grains precipitated during the welding. However, no discernible cracks were in evidence. Figure 22 shows a view of the Ta header after removal of the Ta dome.

Removal of the Tantalum and Type 316SS headers from the Hg inlet was the last step in the boiler disassembly procedure. To separate the headers, the tube bundle was cut at a point such that the remaining plug insert length left in the boiler would be four feet. A front view of the 316SS header is shown in Figure 23. All of the tube-to-header welds were inspected and found to be free of defects. The three pins spaced 120° apart had been installed to prevent the two headers from contacting each other and to prevent over-extension of the tandem bellows. Figure 24 is a photograph of the boiler in its present state.

B. Results of Material Investigation

After the boiler was disassembled, the parts that were removed were metallographically and chemically analyzed and evaluated by the Materials Group (see Appendix A). Listed below are the specific items covered by the Material investigation.

1. 90% Ta - 10% W swirl wire
2. 321SS static NaK tube
3. Tantalum tube under the static NaK tube crack
4. Ta/316SS Hg inlet transition joint
5. Zirconium foil
6. Ta reducer at the Hg inlet
7. Ta tube at Hg inlet
8. Ta orifice and tube at the Hg inlet header

Appendix A of this report was prepared by the Materials Group on their findings. The remainder of this section summarizes results of the investigation of the eight preceding items.

90% Ta - 10% W Swirl Wire

Shown in Appendix A is a two-inch long section of swirl wire that was found in the turbine Hg inlet filter (magnified 20 times) revealing numerous cracks. Metallographic examination of this piece of wire plus two other samples of wire not used in the boiler revealed that bending of the wire alone did not cause cracking and eventual fracture. Treatment of the two sample wires by chemical cleaning per specification AGC 10319/8 resulted in embrittlement as indicated by subsequent bending tests. However, it was found that after exposure at elevated temperatures, hydrogen was outgassed and ductility was restored. On three occasions during boiler operation (including that following the boiler chemical cleaning after 1313 hours) hydrogen was detected in the Hg loop. One source of hydrogen was various oils, that had been detected in the Hg loop.

The failure of the swirl wire was most probably due to the combination of the loss of ductility from hydrogen embrittlement and residual stresses in the wire from the coiling operation.

The wire sample obtained from the TAA filter and a new, unused wire sample were chemically analyzed for carbon, hydrogen, nitrogen, and oxygen. The results in parts per million (ppm) are given below.

	Parts Per Million (PPM)			
Element (in PPM)	C	H	N	O
Ta-10W wire from TA inlet filter	20	36	50	550
Ta-10W wire, new, unused	63	25	100	630

321SS Static NaK Tube

The failure of the 321SS tube occurred at the baffle nearest to the PNL exit port in the transition from round to oval cross-section as shown in Figure 25. Also shown in the figure are the temperatures for transient and steady-state conditions measured during test in PCS-1. The particular temperature profiles shown were chosen as being representative of the many profiles obtained since August 1968. Using these temperature data, Reference 5 states that for transient conditions, the stress was 44,000 psi and cyclic thermal stressing during startup/shutdown cycles was the predominant failure mode (See Appendix B).

A photomicrograph of the failed section of the tube is shown in Appendix A. A metallographic comparison of the failed 321SS with an unfailed tube indicated a normal structure except at the fracture where the structure was austenitic. The percentage of sigma phase present in the normal 321SS structure was less than one percent.

Tantalum Tube Under the Static NaK Tube Cracks

It was observed that severe localized thinning had taken place on the O.D. surface of the tantalum tube directly beneath the 321SS tube crack. From a dimensional stackup of the boiler parts, it was found that the "washed out" area of the tantalum tube would be directly beneath the crack when the boiler was at full operating condition. In the cold condition, it was 0.537 inch upstream of the crack (toward Hg inlet). The maximum depth of the "wash-out" was .0185 inch or 46% of the .040 inch thick wall tantalum tubing. Examination of the surface showed a smooth, blended surface with no difference in hardness and structure from the unthinned surface and no evidence of oxidation.

Tantalum/316SS Tapered, Co-extruded Transition Joint

Appendix A shows a longitudinal section through the maximum debonded area of the Hg inlet transition joint. Reference 6 showed that this type of joint, made with the identical extrusion parameters, would continue to debond to failure in approximately 35 thermal cycles. The transition joint from the boiler had undergone 27 thermal cycles. Debonding had taken place on both the O.D. and I.D. of the joint for lateral distances of 0.323 inches and .012 inches, respectively.

Zirconium Foil

The purpose of the zirconium foil is to primarily gather oxygen from the static NaK to prevent oxidation of the tantalum surfaces, especially, in the region of the transition joint. From Figures 12 and 14, it is evident that the zirconium at the Hg inlet is much more contaminated than that at the Hg outlet. Samples of the foil from both ends of the boiler were chemically analyzed for carbon, hydrogen, nitrogen, and oxygen.

Element (in PPM)	C	H	N	O
	Parts Per Million (PPM)			
Zr foil, new, representing that used in BRDC #2	26	25	150	780
*Zr foil from NaK inlet of BRDC #2				
1) darker outer wrap.	1335	106	1160	19,500
2) inner wrap, shiny one side	311	84	380	4,850
*Zr foil from NaK outlet of BRDC #2	2,414	8,000	50	7,000
Acid exposed Zr foil from NaK inlet BRDC #2	798	184	810	14,160
Acid exposed Zr foil from NaK outlet BRDC #2	170	4,600	50	6,800

*These samples had carbonates on them therefore the C & O would be expected to be higher than if the CO_3 were not present. Also, the carbonate could absorb water, making the H & O higher.

Tantalum Dome

Examination of the tantalum dome and dome-to-tantalum header weld was made which showed the weld to be excellent while the adjacent dome and header O.D. surfaces indicated they had been oxidized. It is postulated that this condition had occurred during the boiler repair at 1313 hours due to oxygen contamination of the inert atmosphere of the portable welding dry-box. It is further postulated that the oxide was removed by the static NaK which confirms the belief that NaK will preferentially remove oxygen from tantalum at elevated temperatures in the range of 1100°F. No examination was made of the Hg outlet dome. However, it is suspected that it would show the same results since a Ta/Ta weld was also made here when extensive boiler repairs were required due to the bellows failure.

Tantalum Tube and Orifices

The tantalum tubing which was removed from the Hg inlet of the boiler along with a section of the plug insert showed no evidence of liquid Hg corrosion or erosion. However, the Hg exposed surface did reveal contamination while the surface contacting the lands of the plug insert were clean. Analysis of the contaminating film indicated 3% carbon, 93% iron and 3% nickel. Oxygen contamination was not detected thus eliminating this as a cause of boiler performance decay. In addition, analysis of one tantalum tube showed the presence of duoseal oil which would cause a boiler pressure drop degradation. The iron and nickel are considered to be mass transfer products.

One of the Hg inlet orifices was sectioned and examined for corrosion, erosion, and mass transfer products. None of these was in evidence. The weld joining the tantalum tube, orifice and header was also examined and found to be of excellent quality with larger grain sizes than that of the parent material (as could be expected). On the downstream (away from Hg Inlet) side of the header the effects of tube bending stresses were evident but are considered inconsequential to the structural integrity of the boiler.

V. CONCLUDING REMARKS AND RECOMMENDATIONS

1. The tandem transition joint at the mercury inlet would have to be redesigned or the co-extrusion process would have to be more closely controlled to ensure a better tantalum-to-stainless steel bond. The short life of this type of joint was borne out by thermal cycling in the Metallurgical Laboratory. If increased life is unattainable, the joint should be replaced by an axially co-extruded bimetal joint of sufficient length or by a rigorously controlled brazed joint similar to the mercury outlet joint.

2. The tantalum -10% tungsten swirl wire should be replaced with a material that is insensitive to hydrogen embrittlement. Although it was found that the cleaning process contributed to the hydrogen embrittlement, it is well known that hydrogen generation can emanate from the reactor and oil contaminants in the mercury loop.

3. It appears that the method of baffling the primary NaK flow at its exit has to be carefully analyzed to preclude excessive axial and circumferential thermal gradients versus axial and circumferential lengths. The cracks in the static NaK tube at the baffle indicate that thermal stress was the predominant failure mode.

4. The two shell failures in the vicinity of the primary NaK outlet are indicative of excessive thermal stresses for the same reason as (3) above. It is conjectured that a third failure may have occurred in this area if the failed shell was removed and replaced with one of similar design. The doubler added sufficient strength to the existing shell to contain the NaK even when the original shell failed.

5. The zirconium foil at the mercury inlet was badly decomposed after 7386 hours of testing. This could have been accelerated by the static NaK leak because of NaK oxides from the primary loop. In any event, the foil should be much thicker than that used in this boiler to preclude fragmentation and transport of pieces to critical areas such as the bellows convolutes and the annuli between the tantalum tubes and their respective static NaK tubes.

6. The deconditioning of the boiler during startups following the first run appears to be predominantly the result of oil and/or other contamination in the mercury loop. Sources of contamination such as the leak detector and the vacuum pumps can be greatly reduced or eliminated by PCS-1 system redesign. The space seal designs of the TAA and mercury PMA should be re-examined with the goal of eliminating these two sources of 4P3E oil, especially during startup and shutdown.

The following problems encountered with earlier boiler designs were solved in the boiler discussed here:

1. Tantalum as the mercury containment material has shown it to be superior to 9% chrome, 1% moly steel used previously. The tantalum has good wetting characteristics in mercury and has a greater recovery after contamination. No cracking, erosion, or corrosion could be found in the areas examined.

2. This boiler was more stable than previous boilers even at extreme off-design conditions.

3. The principle of double containment was proven in this design and testing continued with no deleterious effects, irrespective of the failure of the static NaK tube. A single failure between the primary NaK and the mercury automatically terminated testing in earlier designs.

LIST OF REFERENCES

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Subject: Evaluation of Boiler Primary-Static NaK Leak

3. Memorandum 5351:69:0155, J. N. Hodgson dtd 15 April 1969
Subject: Repeat Test on Size of Primary-Static NaK Leak in PCS-1 Boiler

4. Memorandum 5351:68:0196, J. N. Hodgson dtd 29 May 1968
Subject: Startup of Run D-43-6-22, PCS-1 Phase IV, Step 3

5. Stress Analysis No. SA-B-240, S. Krikopulo, dtd 2 December 1969
Subject: Crack on the Static NaK Oval Tube

6. TM 4923:69:579, H. E. Bleil dtd 1 May 1969
Subject: Thermal Exposure Evaluation of Tantalum/316SS Transition Joints

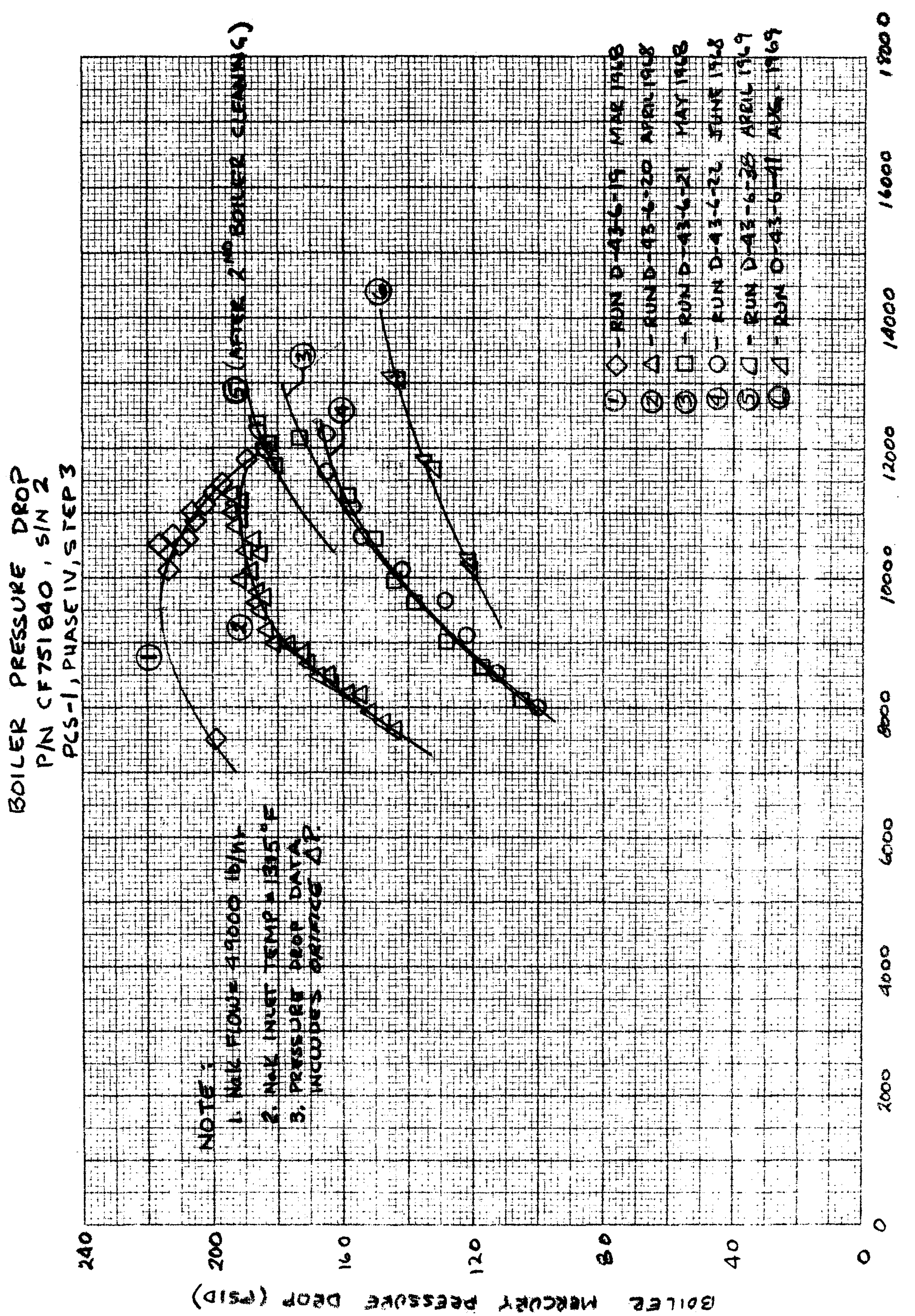
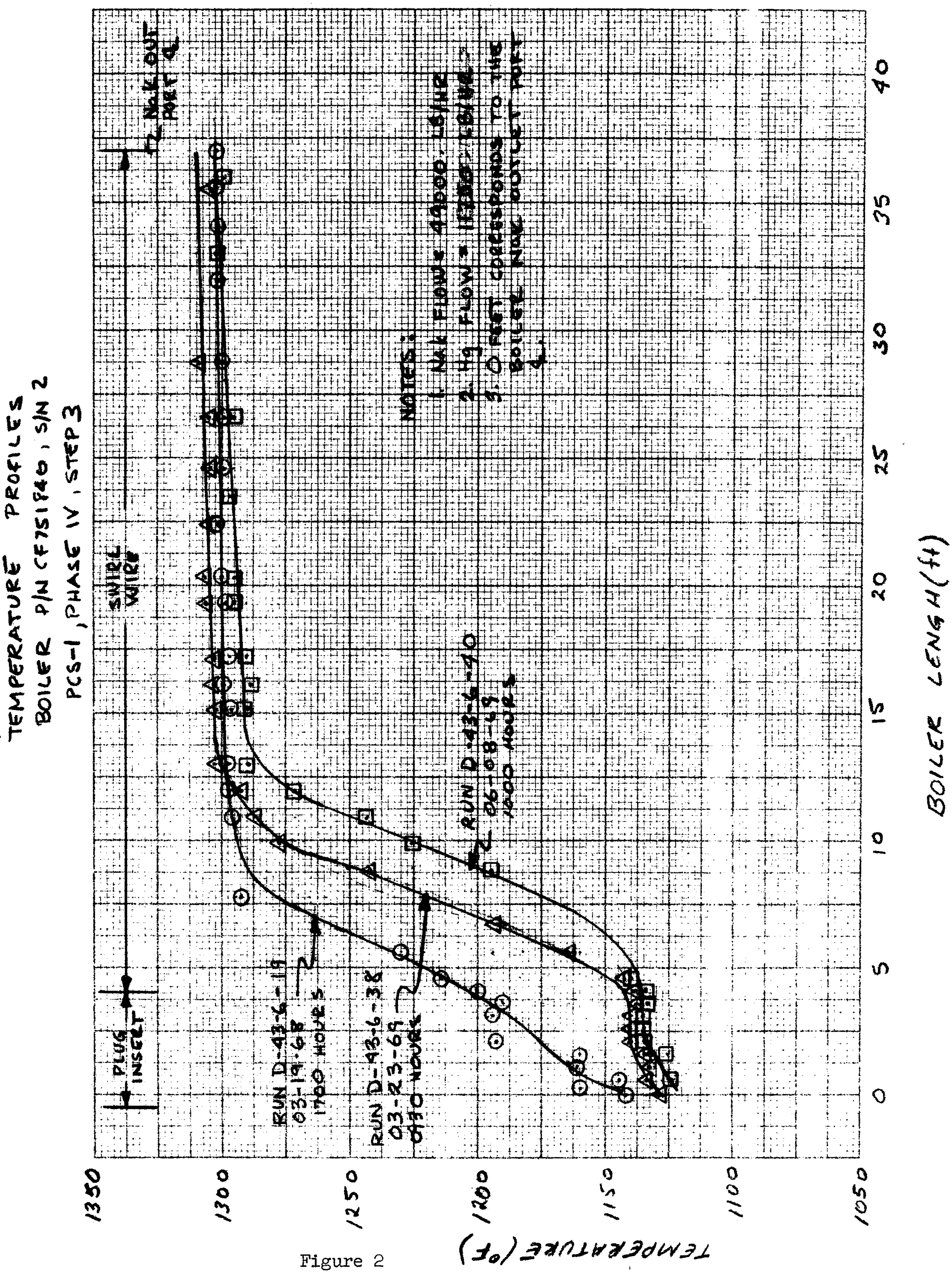

BOILER PRESSURE DROP
P/N CF751840, S/N 2
PCS-1, PHASE IV, STEP 3
NOTE:
1. NaK FLOW = 49000 lb/hr
2. NaK INLET TEMP = 1335°F
3. PRESSURE DROP DATA INCLUDES ORIFICE ΔP.
(AFTER 2ND BOILER CLEANING)
1 - RUN D-43-6-19 MAR 1968
2 - RUN D-43-6-20 APRIL 1968
3 - RUN D-43-6-21 MAY 1968
4 - RUN D-43-6-22 JUNE 1968
5 - RUN D-43-6-38 APRIL 1969
6 - RUN D-43-6-41 AUG. 1969
BOILER MERCURY PRESSURE DROP (PSID)
MERCURY LIQUID FLOW (LB/HR)
0
40
80
120
160
200
240
0
2000
4000
6000
8000
10000
12000
14000
16000
18000

Figure 1



TEMPERATURE PROFILES
BOILER P/N CF751F40, S/N 2
PCS-1, PHASE IV, STEP 3
PLUG INSERT
SWIRL WIRE
NaK OUT PORT 4
NOTES:
1. NaK FLOW = 49000 LB/HR
3. 0 FEET CORRESPONDS TO THE BOILER NaK OUTLET PORT 4.
RUN D-43-6-19
03-19-68
1700 HOURS
RUN D-43-6-38
03-23-69
RUN D-43-6-40
06-08-69
1000 HOURS
BOILER LENGH (ft)
TEMPERATURE (°F)
1350
1300
1250
1200
1150
1100
1050
0
5
10
15
20
25
30
35
40

Figure 2

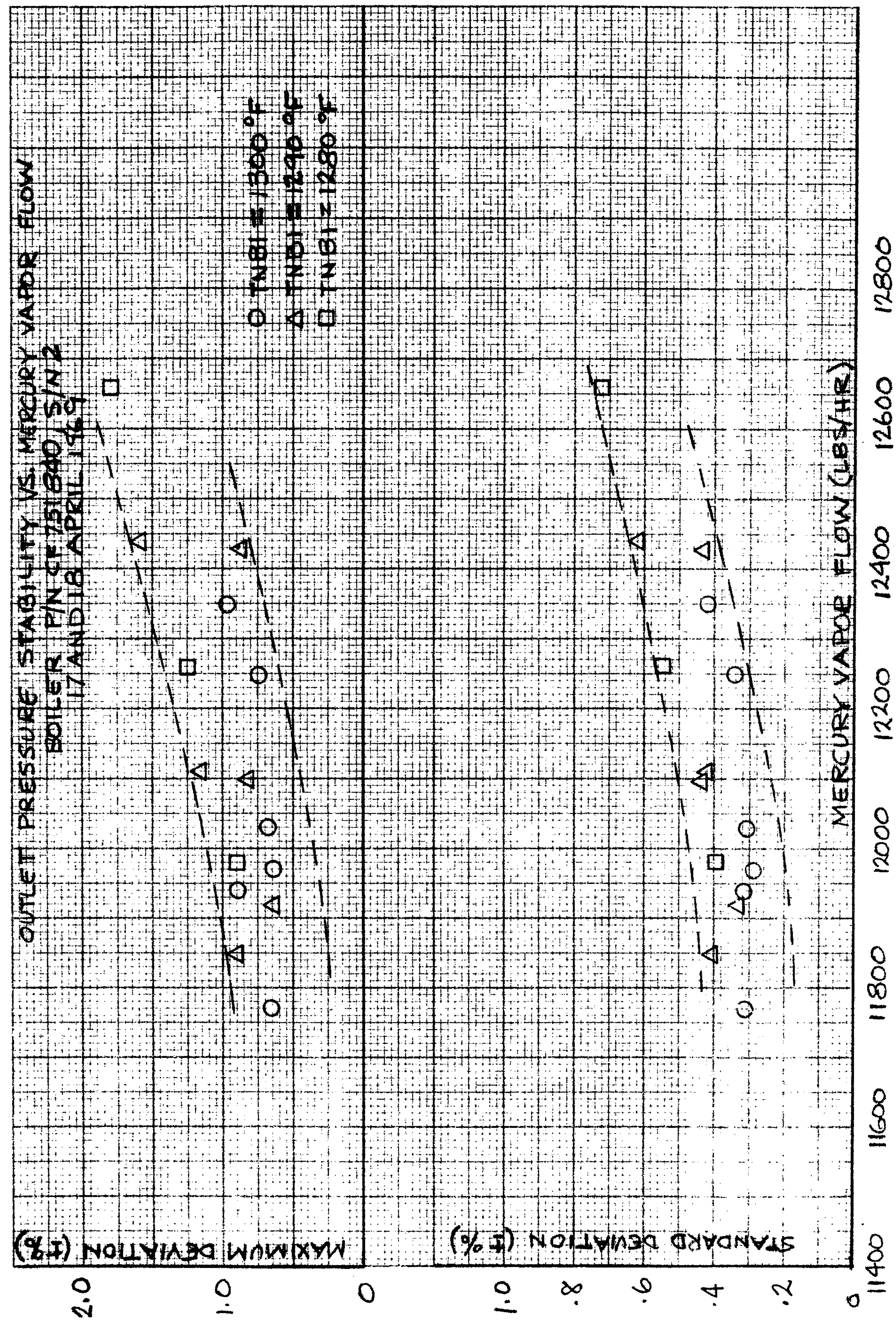
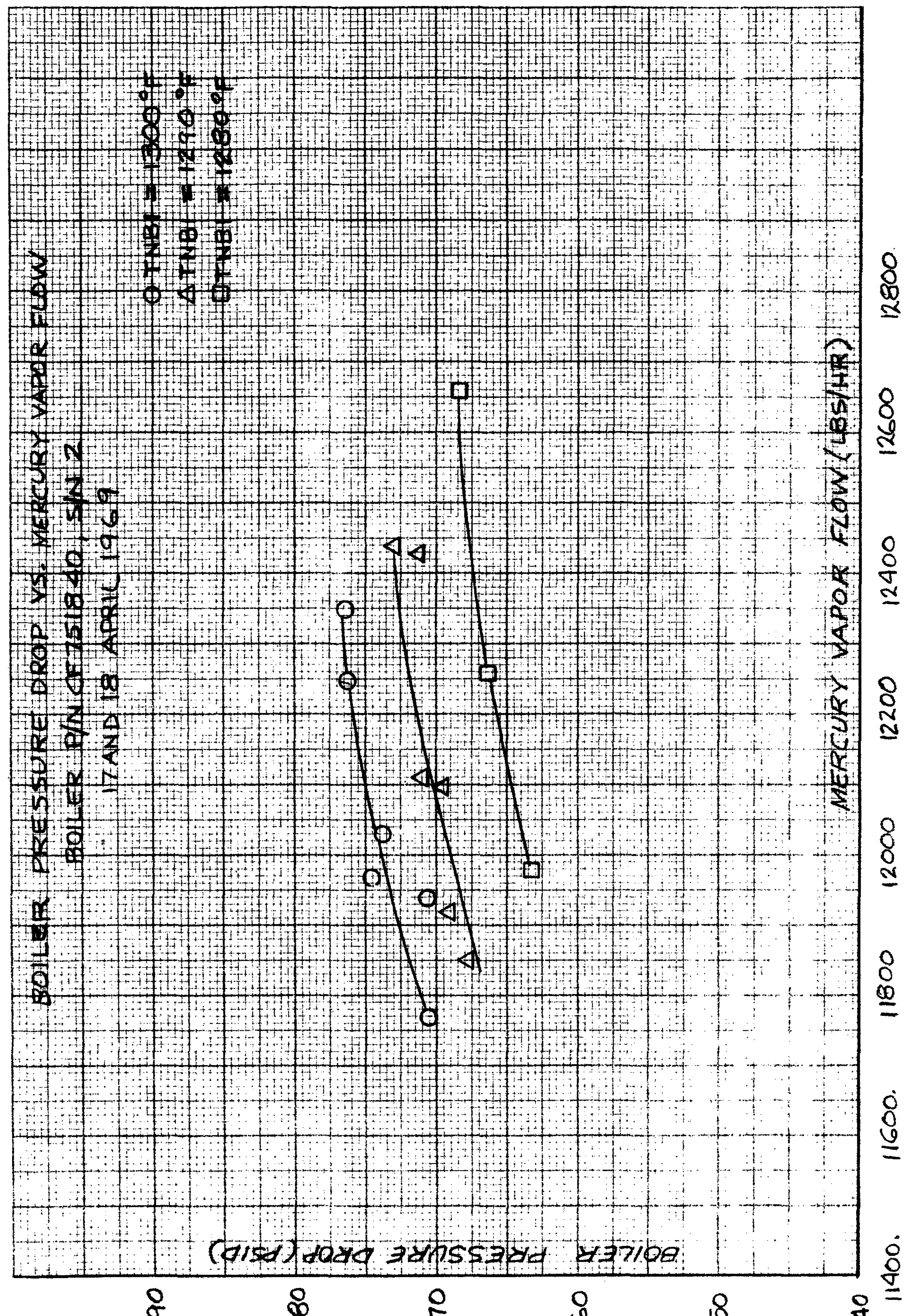

OUTLET PRESSURE STABILITY VS. MERCURY VAPOR FLOW
BOILER P/N CF751840 S/N 2
17 AND 18 APRIL 1969
○ TN81 = 1300 °F
△ TN81 = 1290 °F
□ TN81 ≈ 1280 °F
MAXIMUM DEVIATION (±%)
STANDARD DEVIATION (± %)
MERCURY VAPOR FLOW (LBS/HR)
2.0
1.0
0
1.0
.8
.6
.4
.2
0
11400
11600
11800
12000
12200
12400
12600
12800

Figure 3



BOILER PRESSURE DROP VS. MERCURY VAPOR FLOW
BOILER P/N CF751840, S/N 2
17 AND 18 APRIL 1969
○ TNBI = 1300°F
△ TNBI = 1290°F
□ TNBI = 1280°F
MERCURY VAPOR FLOW (LBS/HR)
BOILER PRESSURE DROP (PSID)
11400.
11600.
11800
12000
12200
12400
12600
12800
140
150
160
170
180
190

Figure 4

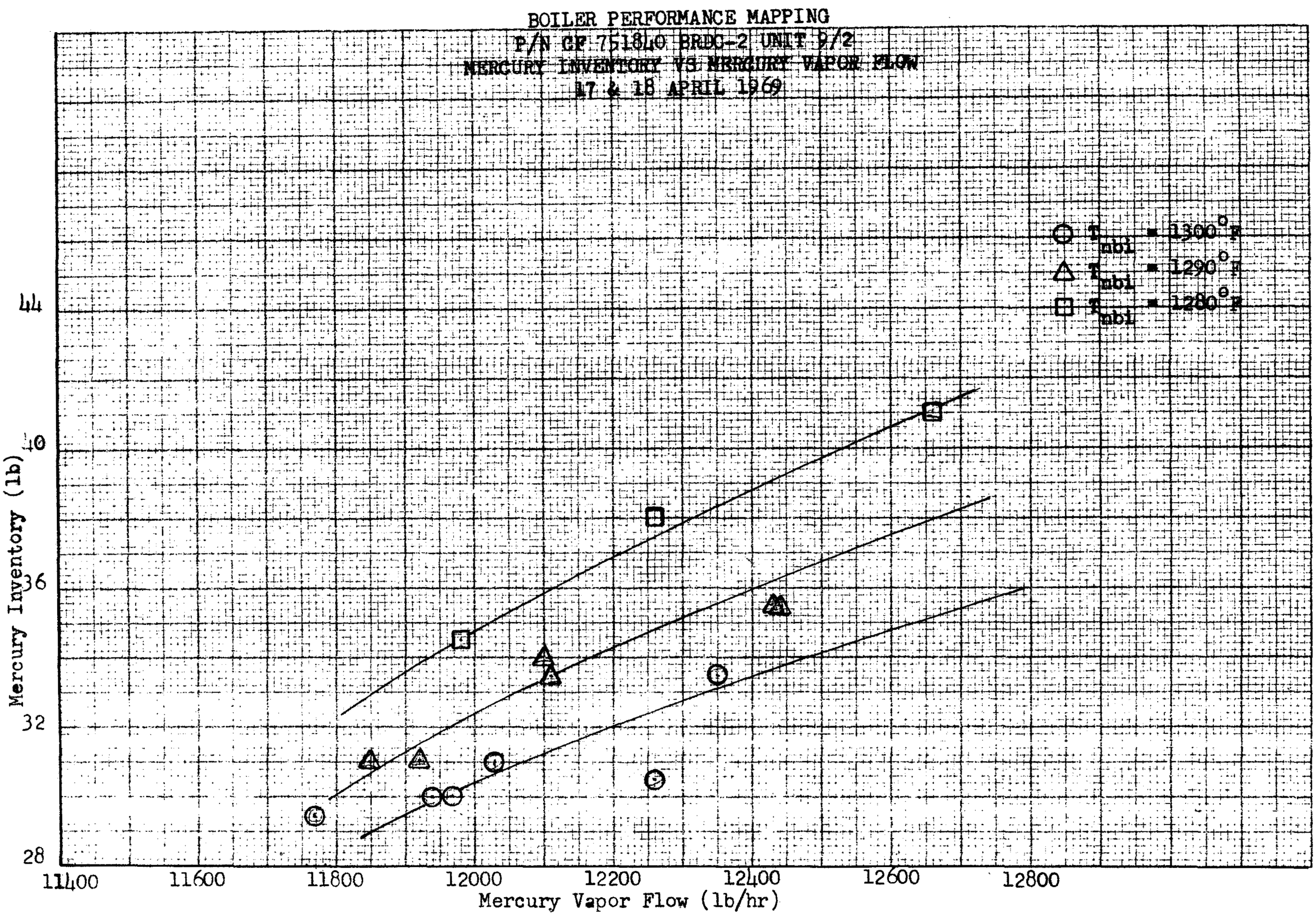

BOILER PERFORMANCE MAPPING
P/N CF 751840 BRDC-2 UNIT 9/2
MERCURY INVENTORY VS MERCURY VAPOR FLOW
17 & 18 APRIL 1969
T_{mbi} = 1300° F
T_{mbi} = 1290° F
T_{mbi} = 1280° F
Mercury Inventory (lb)
Mercury Vapor Flow (lb/hr)
28
32
36
40
44
11400
11600
11800
12000
12200
12400
12600
12800

Figure 5

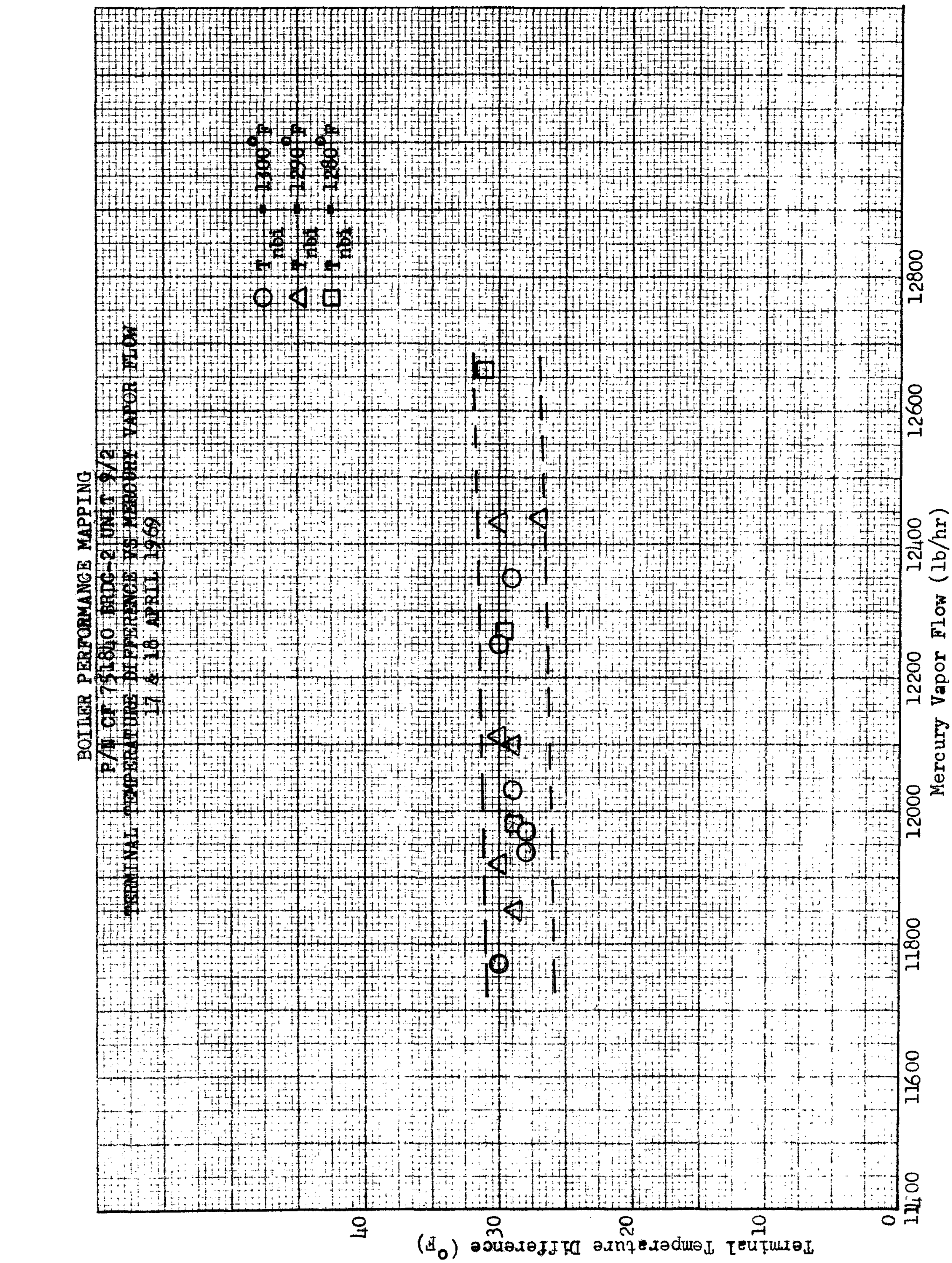
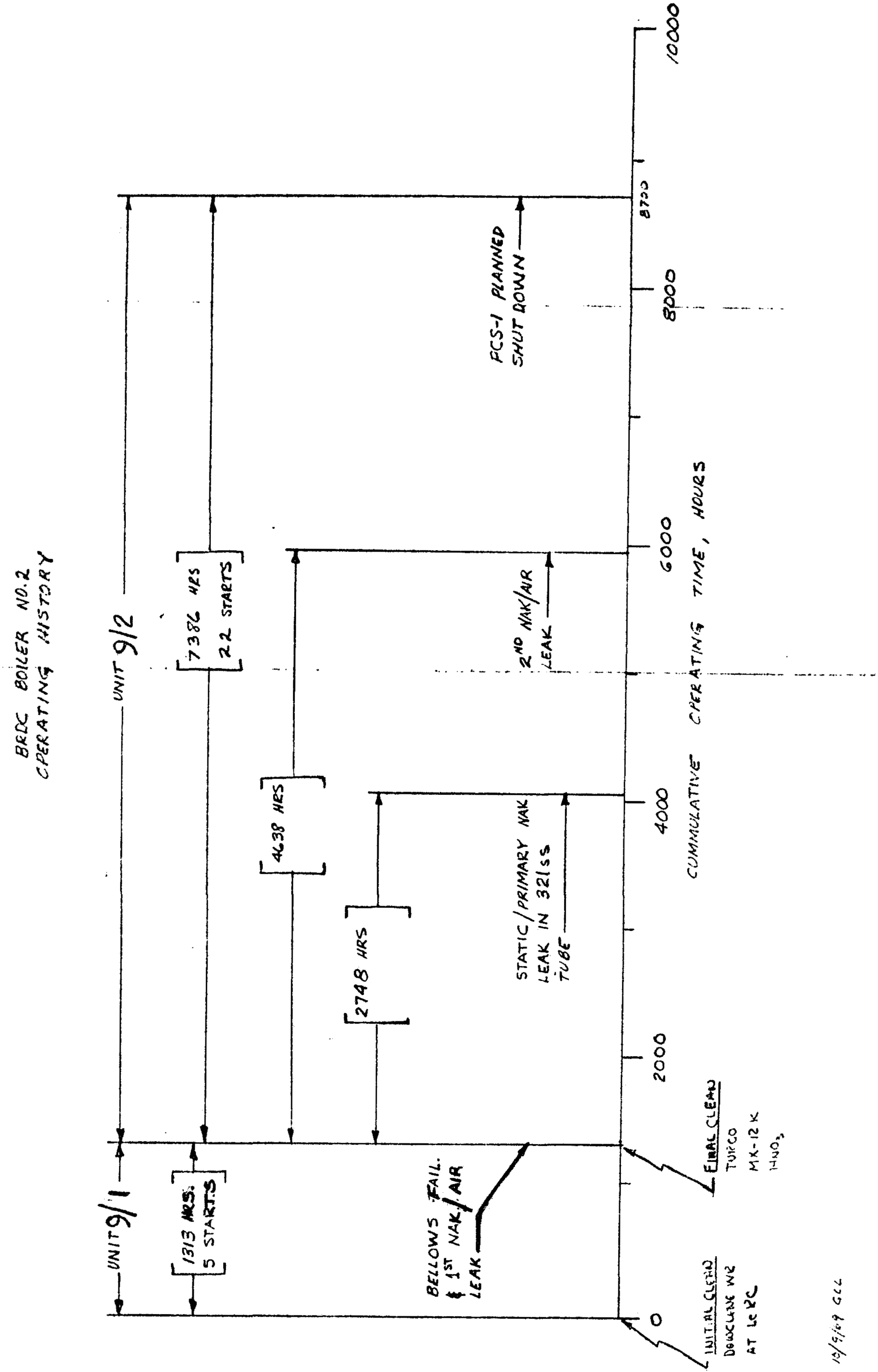
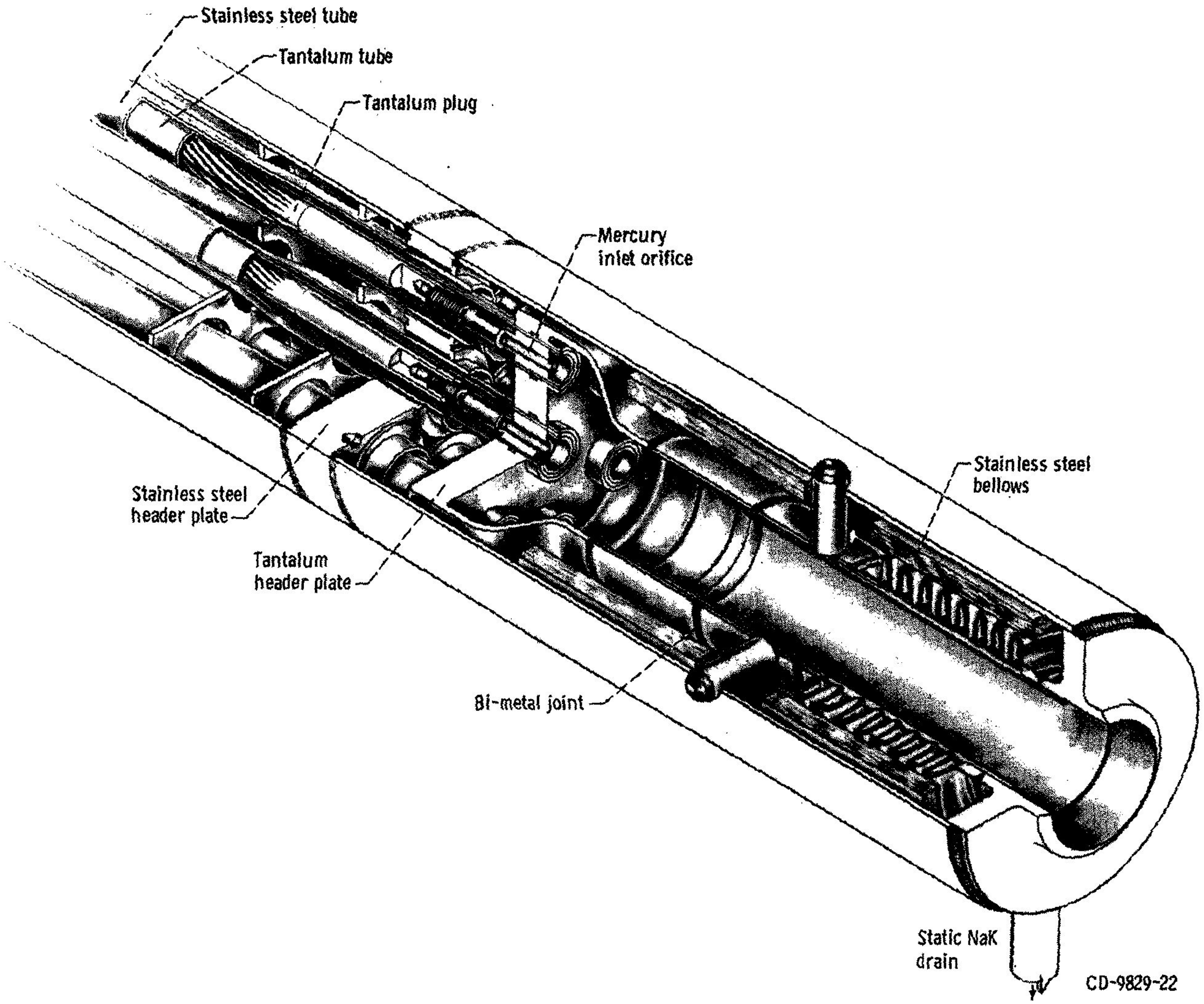

BOILER PERFORMANCE MAPPING
P/N CF 751640 BRDC-2 UNIT 9/2
TERMINAL TEMPERATURE DIFFERENCE VS MERCURY VAPOR FLOW
17 & 18 APRIL 1969
T nbi = 1300°F
T nbi = 1250°F
T nbi = 1200°F
Terminal Temperature Difference (°F)
0
10
20
30
40
Mercury Vapor Flow (lb/hr)
11400
11600
11800
12000
12200
12400
12600
12800

Figure 6



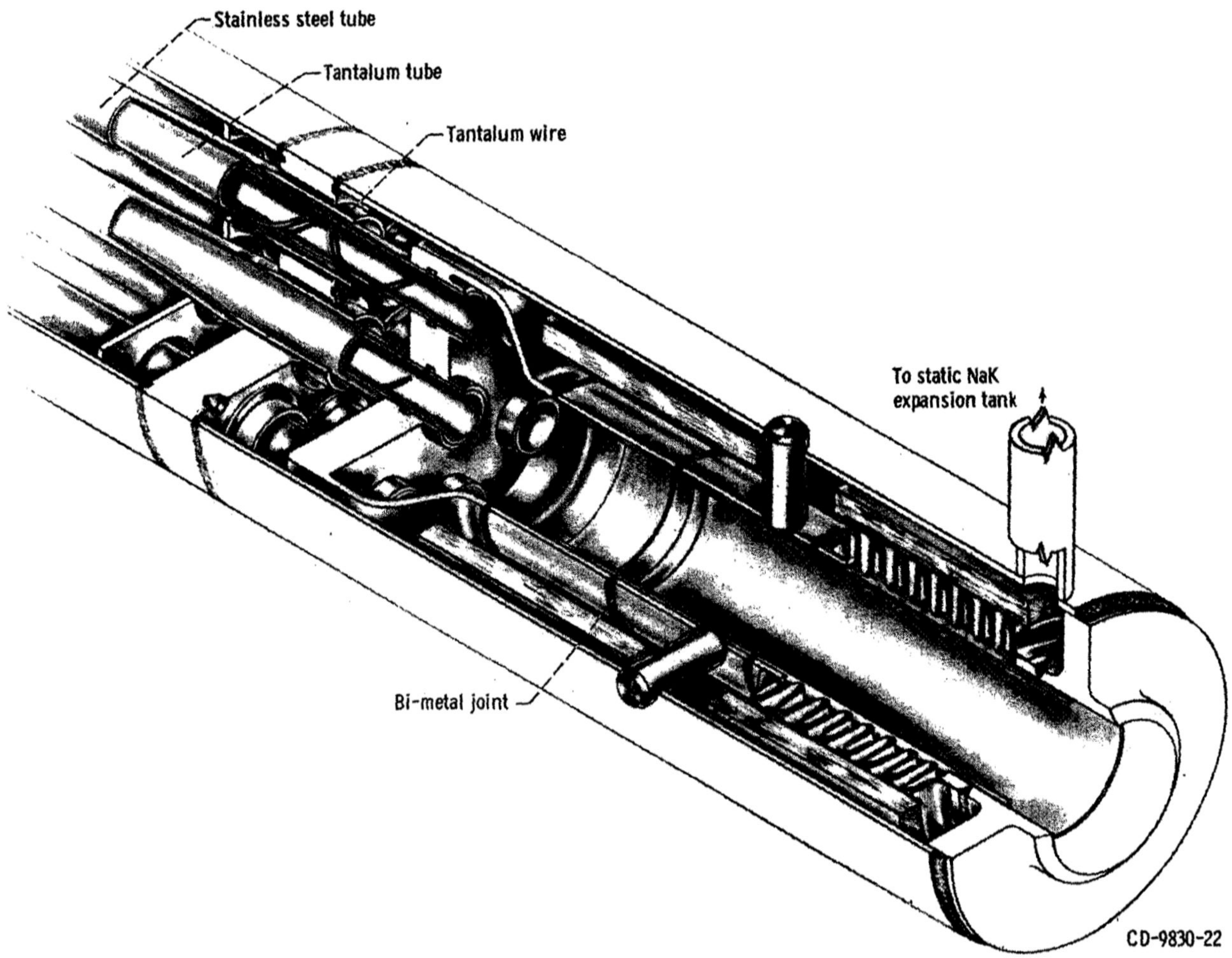
BRDC BOILER NO. 2
OPERATING HISTORY
UNIT 9/1
UNIT 9/2
1313 HRS. 5 STARTS
7386 HRS 22 STARTS
4638 HRS
2748 HRS
BELLOWS FAIL. & 1ST NAK/AIR LEAK
STATIC/PRIMARY NAK LEAK IN 321 SS TUBE
2ND NAK/AIR LEAK
PCS-1 PLANNED SHUT DOWN
0
2000
4000
6000
8000
8700
10000
COMMULATIVE OPERATING TIME, HOURS
INITIAL CLEAN
DOWCLENE WR
AT LeRC
FINAL CLEAN
TURCO
MK-12K
HNO3
10/9/69 GLL

Figure 7



Mercury Inlet Section - Taken from NASA TM X-52452

Figure 8a



Mercury Outlet Section - Taken from NASA TM-X 52452

Figure 8b

NOTES: 1. REMOVE ALL BURRS AND SHARP EDGES

2. MACHINED SURFACES TO BE 125√ UNLESS OTHERWISE SPECIFIED.

3. GAS TUNGSTEN ARC WELD PER AGC-10227/2 USING ITEM 17 WELD ROD.

4. PENETRANT INSPECT WELDS PER MIL-I-6866, TYPE II, METHOD B. PROPAGATING DEFECTS ARE CAUSE FOR REJECTION.

5. RADIOGRAPHICALLY INSPECT WELDS PER MIL-STD-453 AND INTERPRET PER AGC-13860, CLASS 15.

6. HELIUM LEAK TEST PER AGC-STD-1283, METHOD 6.

7. MARK PARTS PER ASD5215C WITH 1266057-(APPLICABLE DASH NUMBER)(APPLICABLE REVISION LETTER) AND PACKAGE PER AGC-13876.

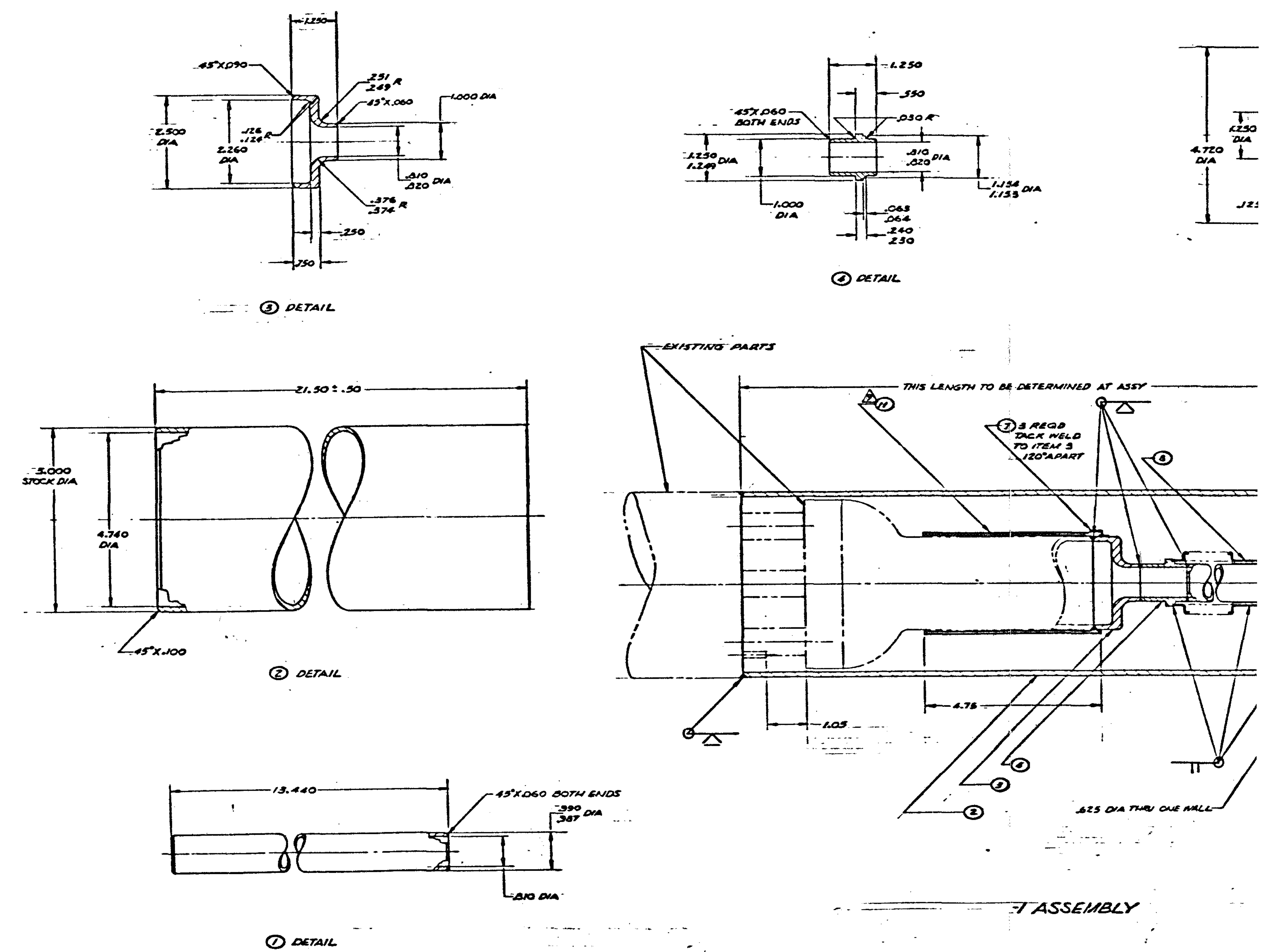
8. MARK PACKAGE PER ASD5215N WITH 1266057-1.

9. FORM FOUR (4) LAYERS OF ITEM 11 AROUND O.D. OF TUBE AS SHOWN.

10. THESE ITEMS ARE USED TO MODIFY THE MERCURY INLET OF NASA BOILER #CF751840/SERIAL #2.

11. CLEAN INDIVIDUAL PARTS PER AGC-STD-1272, METHOD 101, PROCEDURE 2.

12. CLEAN ASSEMBLY PER INSTRUCTIONS OF THE SNAP-8 MATERIALS GROUP.



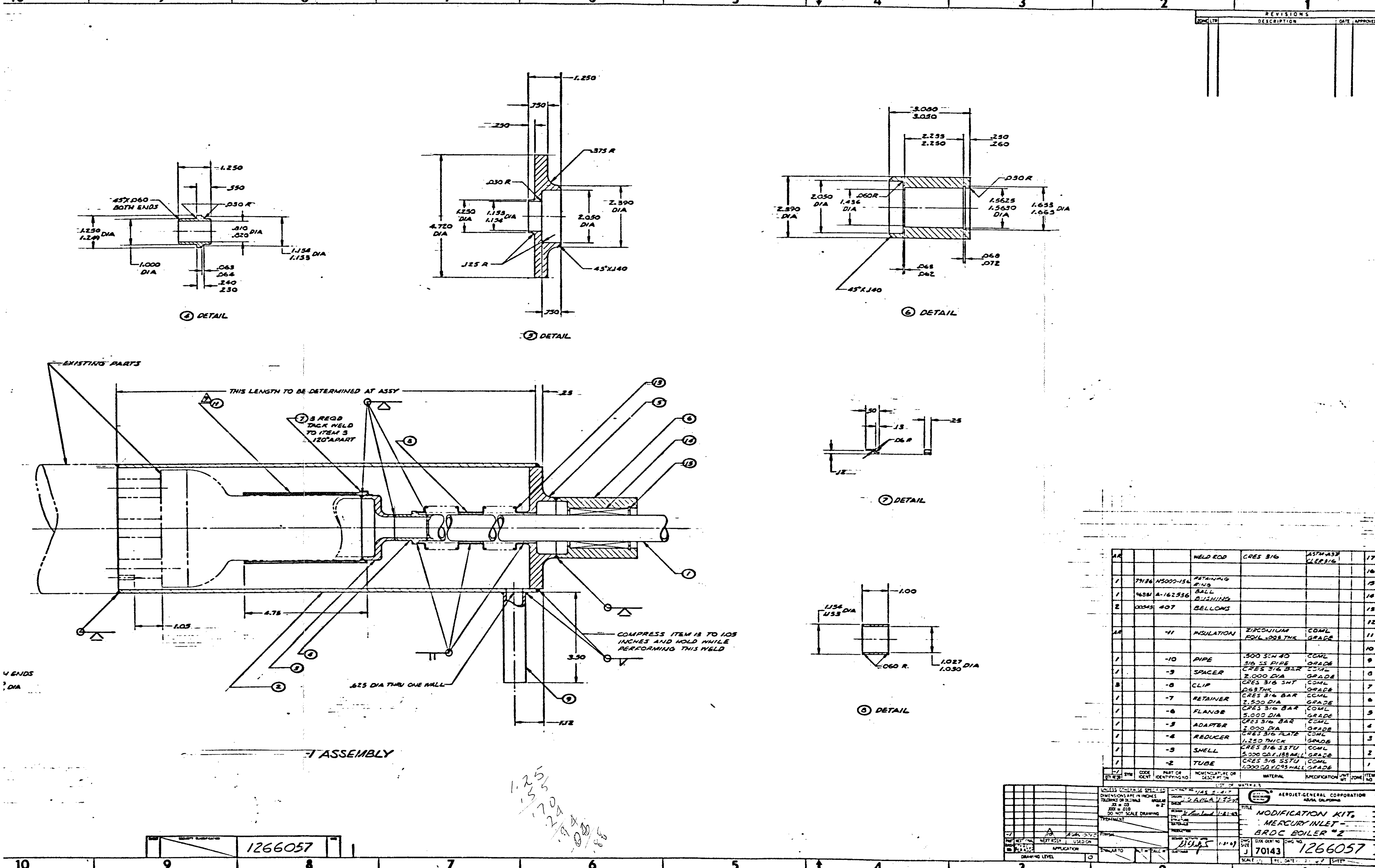

REVISIONS
④ DETAIL
⑤ DETAIL
⑥ DETAIL
⑦ DETAIL
⑧ DETAIL
EXISTING PARTS
THIS LENGTH TO BE DETERMINED AT ASSY
⑦ 3 REQD TACK WELD TO ITEM 3 120° APART
COMPRESS ITEM 13 TO 1.05 INCHES AND HOLD WHILE PERFORMING THIS WELD
.625 DIA THRU ONE WALL
-1 ASSEMBLY
45°X.060 BOTH ENDS
WELD ROD CRES 316 ASTM-A371 CLER316
RETAINING RING
BALL BUSHING
BELLOWS
INSULATION ZIRCONIUM FOIL .005 THK COML GRADE
PIPE .500 SCH 40 316 SS PIPE COML GRADE
SPACER CRES 316 BAR 2.000 DIA COML GRADE
CLIP CRES 316 SHT .063 THK COML GRADE
RETAINER CRES 316 BAR 2.500 DIA COML GRADE
FLANGE CRES 316 BAR 5.000 DIA COML GRADE
ADAPTER CRES 316 BAR 2.000 DIA COML GRADE
REDUCER CRES 316 PLATE 1.250 THICK COML GRADE
SHELL CRES 316 SSTU 5.000 OD X .188 WALL COML GRADE
TUBE CRES 316 SSTU 1.000 OD X .095 WALL COML GRADE
AEROJET-GENERAL CORPORATION AZUSA, CALIFORNIA
MODIFICATION KIT, MERCURY INLET - BRDC BOILER #2
70143
1266057

Figure 9

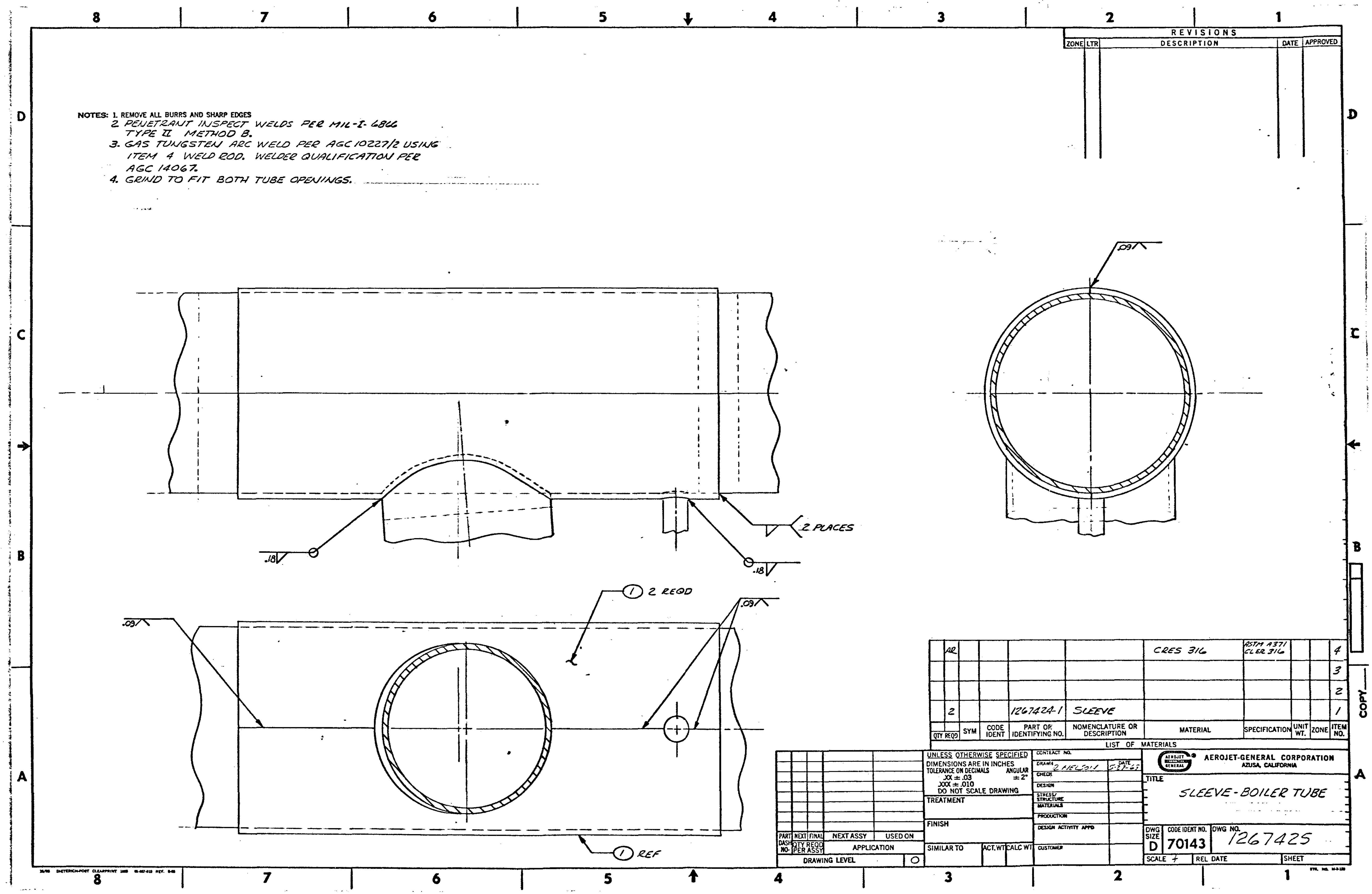
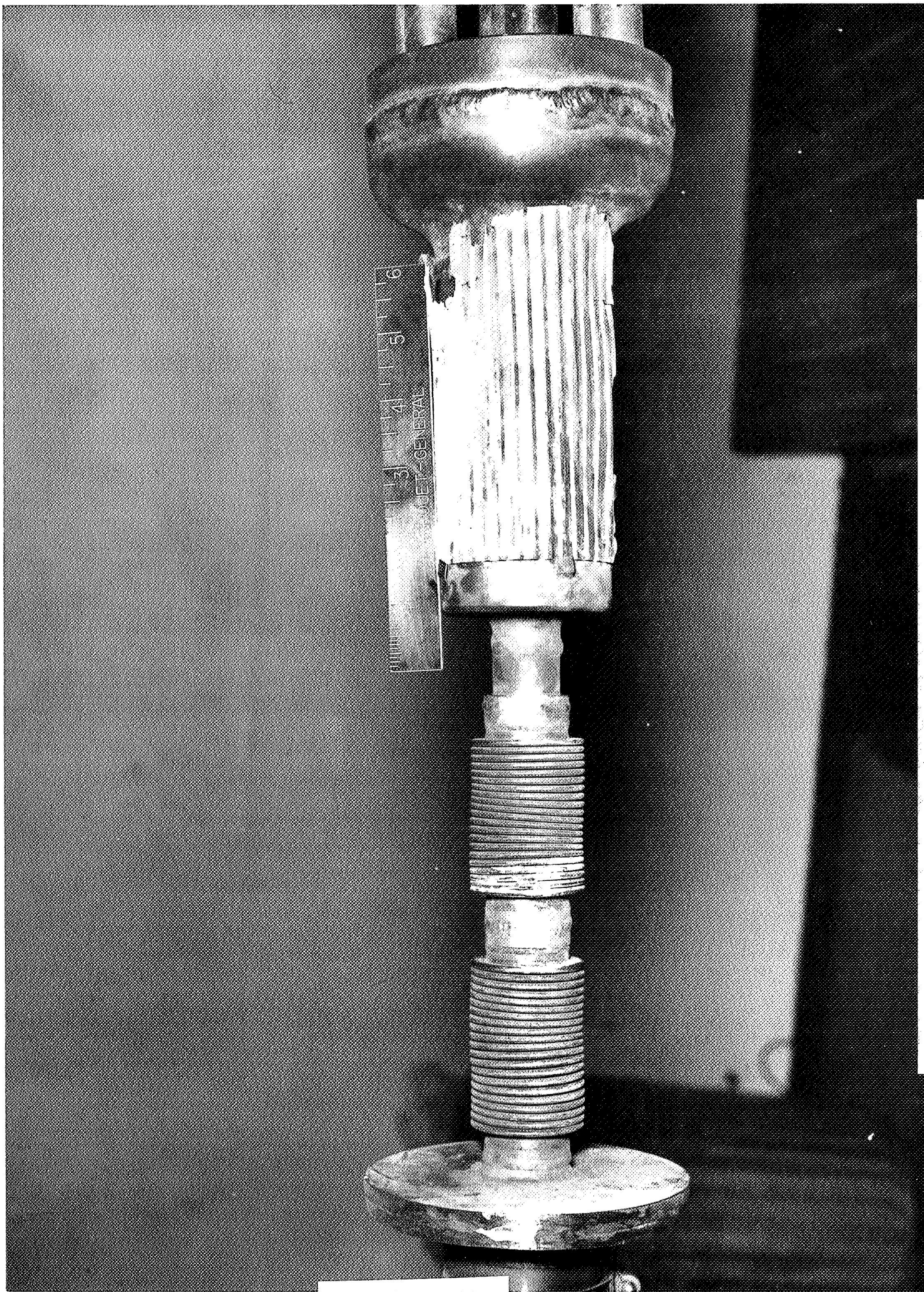

NOTES: 1. REMOVE ALL BURRS AND SHARP EDGES
2. PENETRANT INSPECT WELDS PER MIL-I-6866 TYPE II METHOD B.
3. GAS TUNGSTEN ARC WELD PER AGC 10227/2 USING ITEM 4 WELD ROD. WELDER QUALIFICATION PER AGC 14067.
4. GRIND TO FIT BOTH TUBE OPENINGS.
REVISIONS
ZONE LTR DESCRIPTION DATE APPROVED
2 PLACES
.18
.09
1 2 REQD
1 REF
CRES 316
ASTM A371 CLER 316
2 1267424-1 SLEEVE
QTY REQD SYM CODE IDENT PART OR IDENTIFYING NO. NOMENCLATURE OR DESCRIPTION MATERIAL SPECIFICATION UNIT WT. ZONE ITEM NO.
LIST OF MATERIALS
UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES TOLERANCE ON DECIMALS .XX ± .03 .XXX ± .010 ANGULAR ± 2° DO NOT SCALE DRAWING
TREATMENT
FINISH
SIMILAR TO ACT. WT CALC WT
AEROJET-GENERAL CORPORATION AZUSA, CALIFORNIA
TITLE SLEEVE-BOILER TUBE
DWG SIZE D CODE IDENT NO. 70143 DWG NO. 1267425
SCALE REL DATE SHEET
PART DASH NO. NEXT QTY REQD FINAL PER ASSY NEXT ASSY USED ON APPLICATION
DRAWING LEVEL
COPY

Figure 10

VIEW OF STATIC NAK CONTAINMENT TUBE TAKEN THROUGH
NAK OUTLET PORT OF BOILER P/N CF751840, S/N 2
B/U 9/2 TESTED IN PCS-1 FOR 7386 HOURS, 29 SEPTEMBER 1969

Figure 11

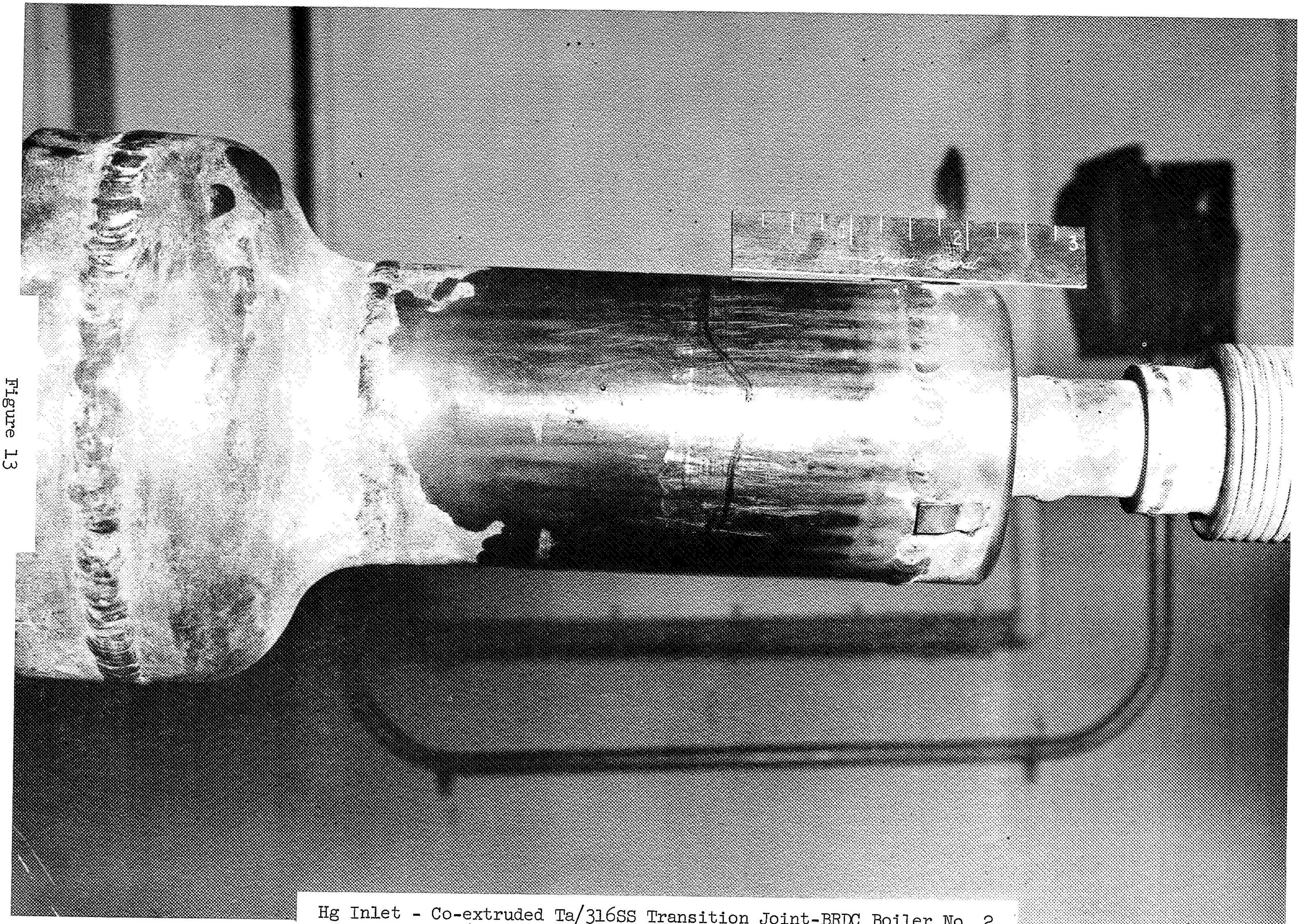


BRDC Boiler No. 2, Hg Inlet Assembly (Ref: 1266057). Operating Time 7386 Hours

1069-120

Figure 12

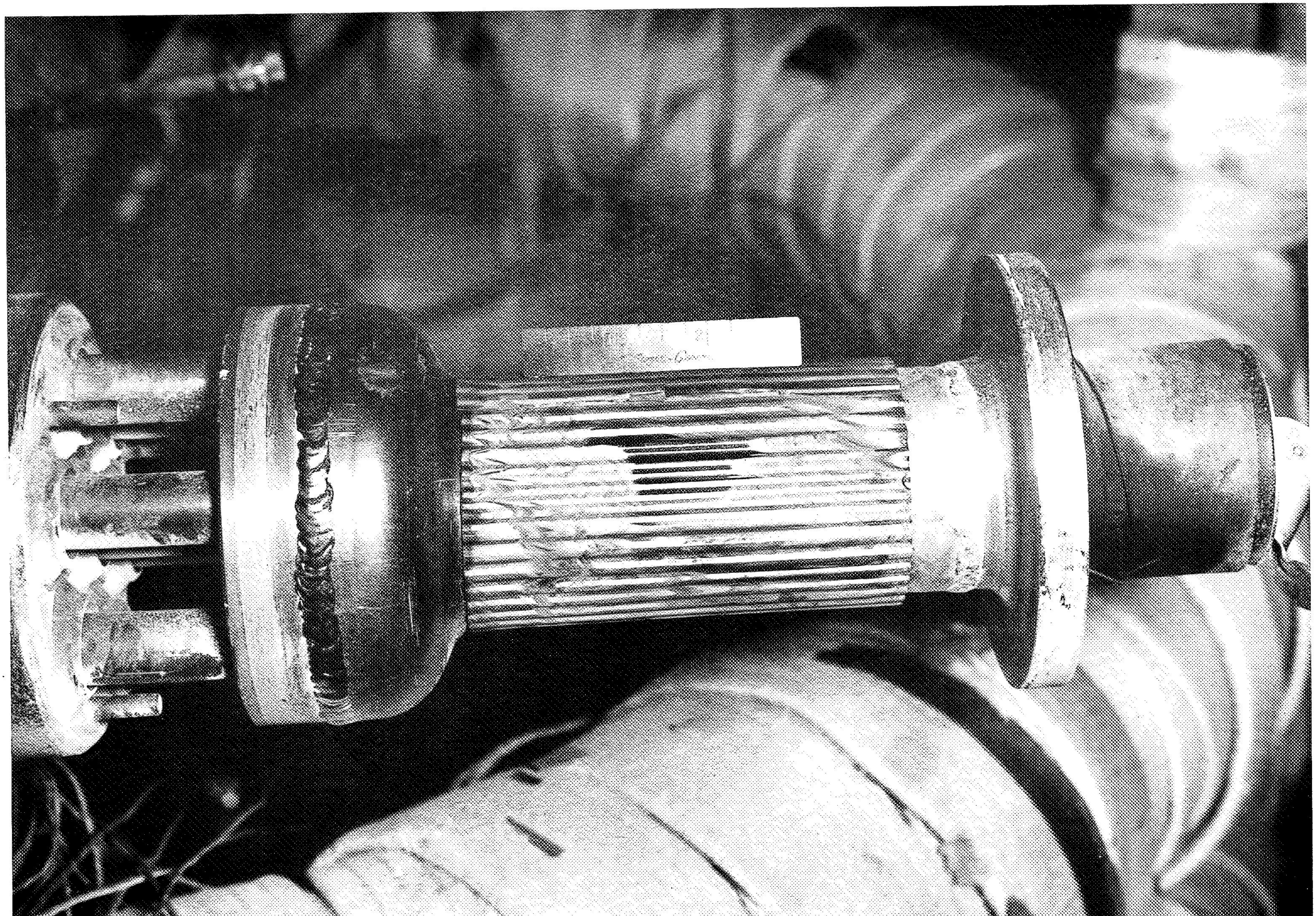
1069-123



Hg Inlet - Co-extruded Ta/316SS Transition Joint-BRDC Boiler No. 2
P/N CF 751840 - After 7386 Hrs. Operation

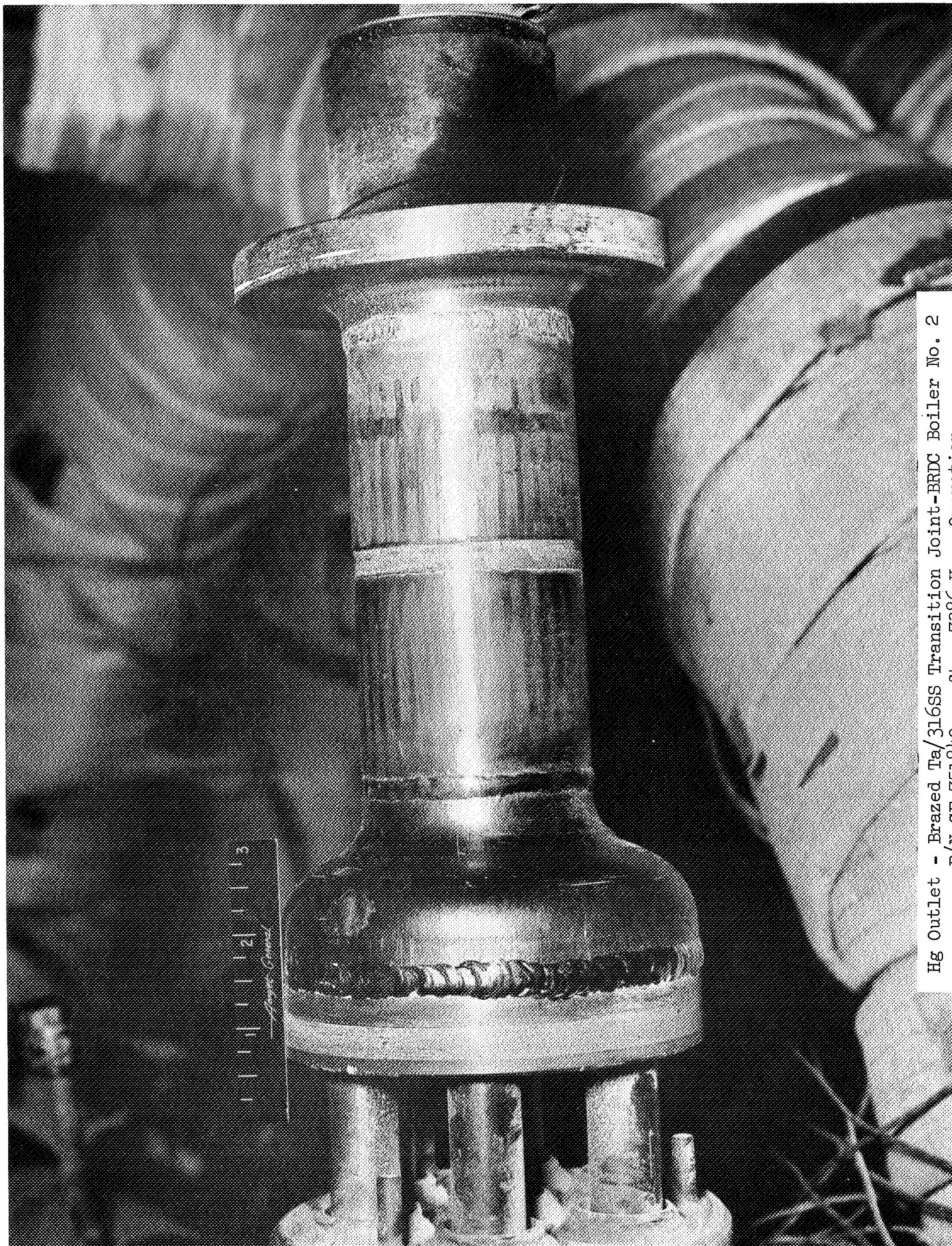
Figure 13

1069-259



Hg Outlet - Brazed Ta/316SS Transition Joint w/Zirconium
Foil-BRDC Boiler No. 2, P/N CF 751840 - After 7386 Hours Operation

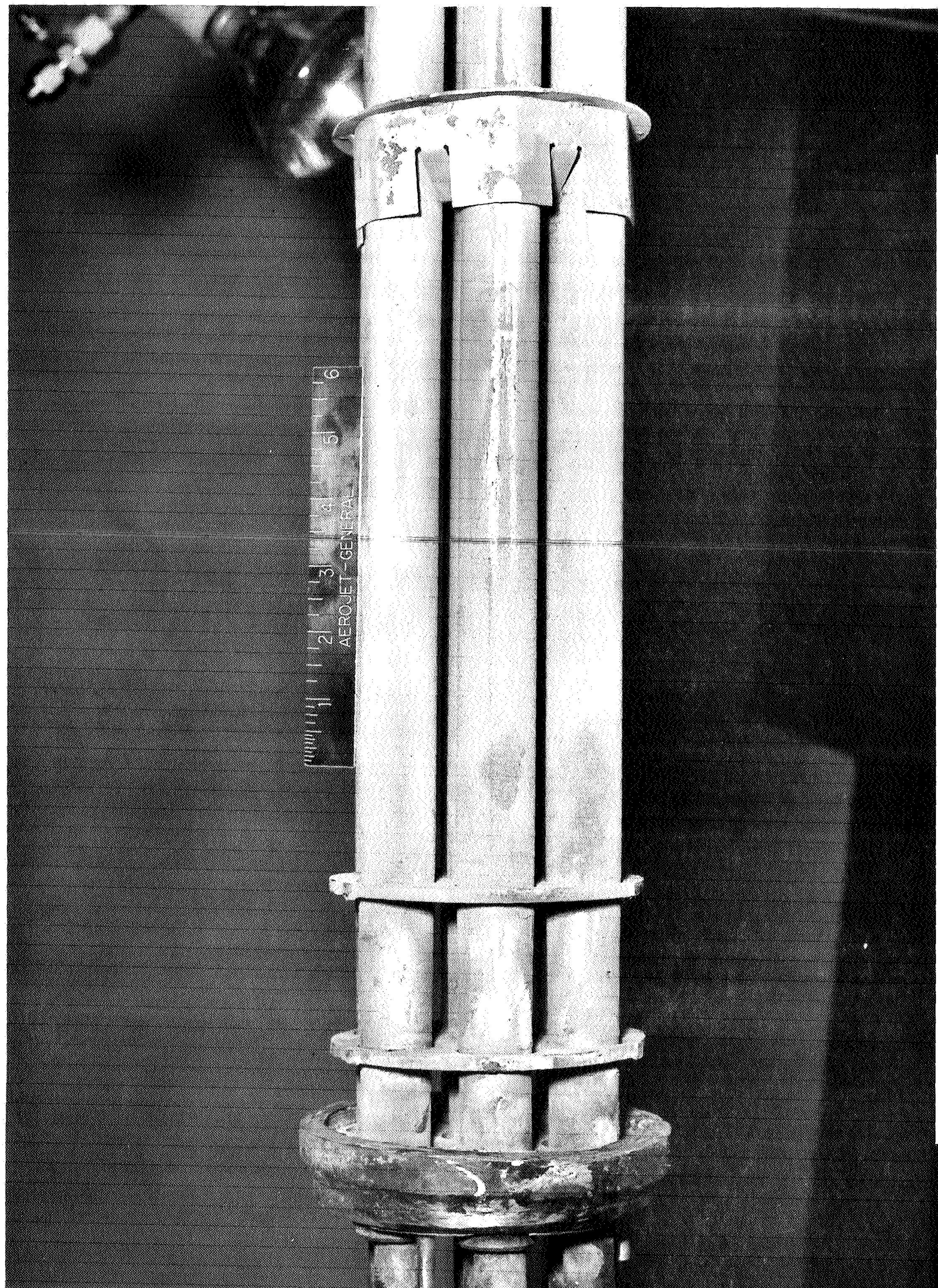
Figure 14



Hg Outlet - Brazed Ta/316SS Transition Joint-BRDC Boiler No. 2
P/N CF 751840 - after 7386 Hrs. Operation

1069-258

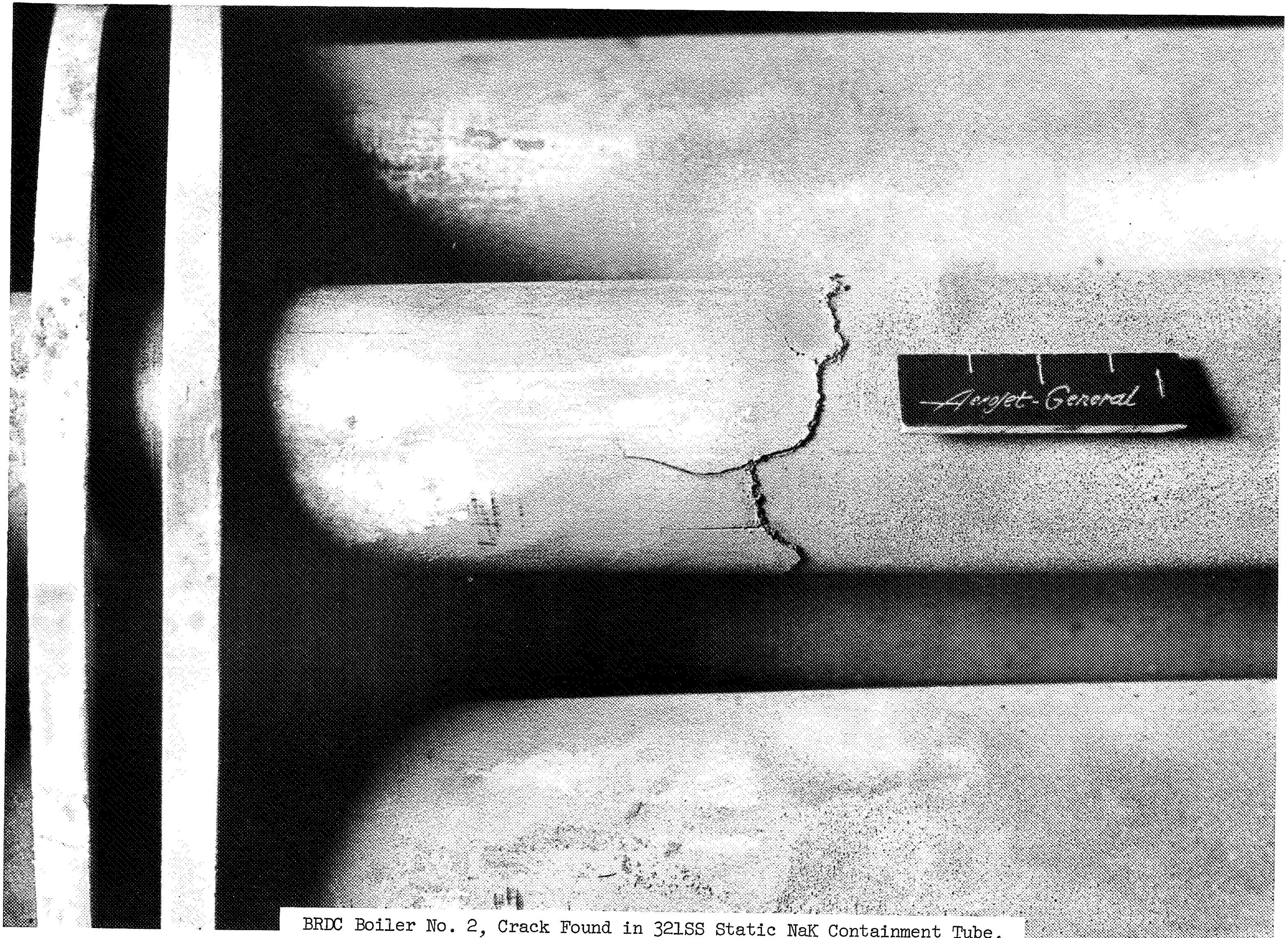
Figure 15



BRDC Boiler No. 2, Tube Bundle Section at NaK Outlet. Operating Time 4061 Hours to Failure

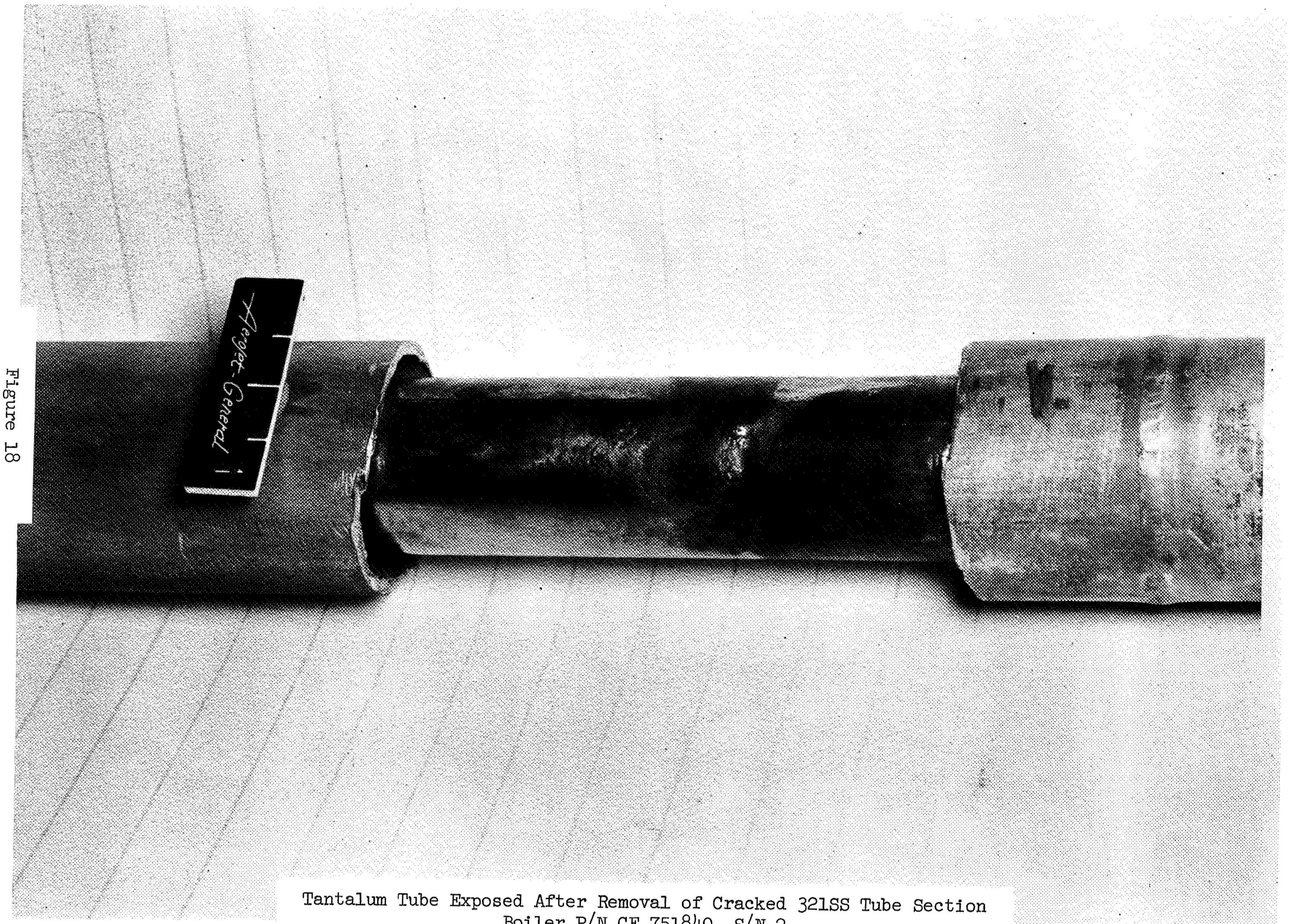
Figure 16

1069-122



BRDC Boiler No. 2, Crack Found in 321SS Static NaK Containment Tube.
Operating Time to Failure 4061 Hours

Figure 17



Tantalum Tube Exposed After Removal of Cracked 321SS Tube Section
Boiler P/N CF 751840, S/N 2

Figure 18

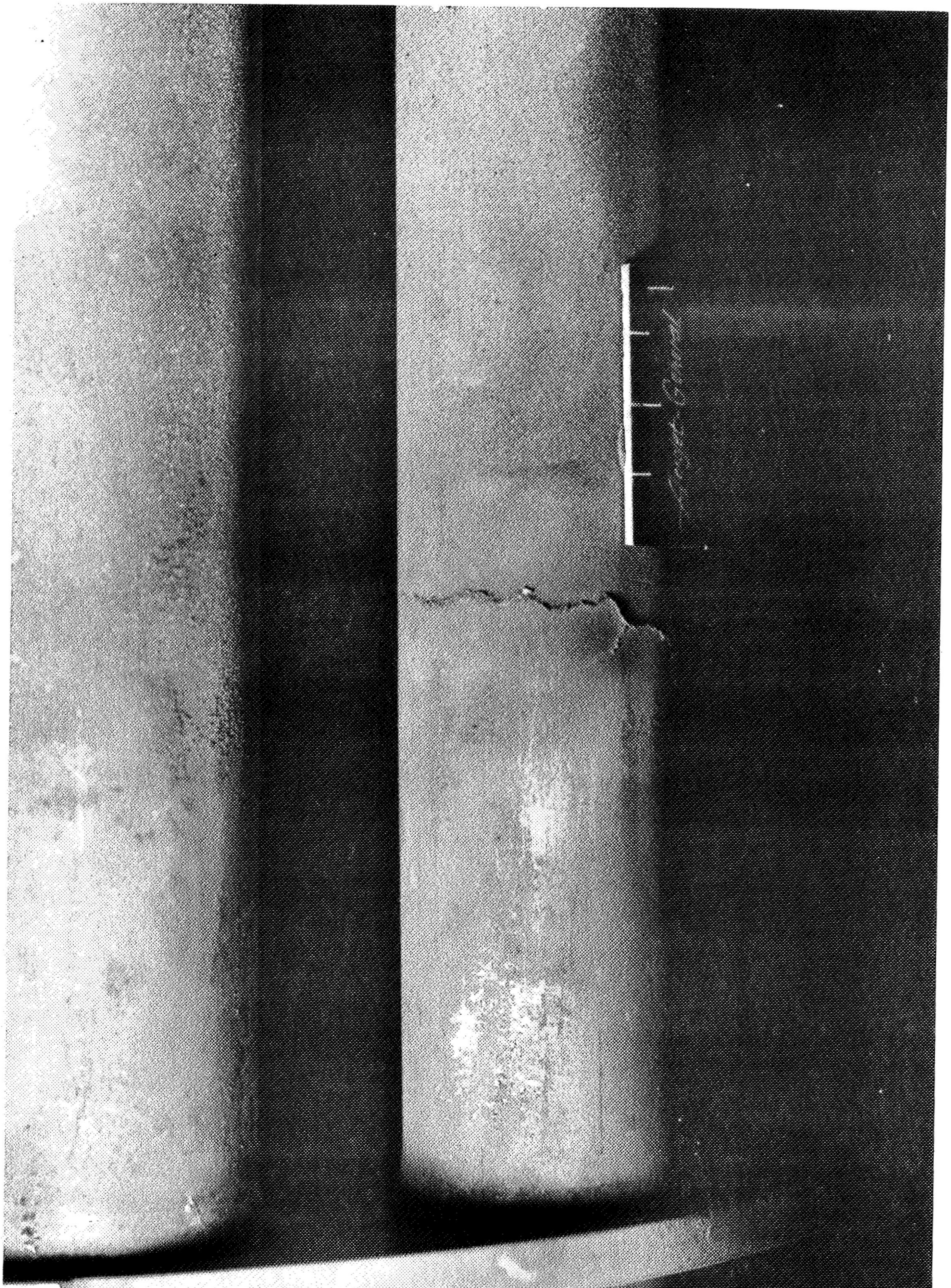


FIG 19

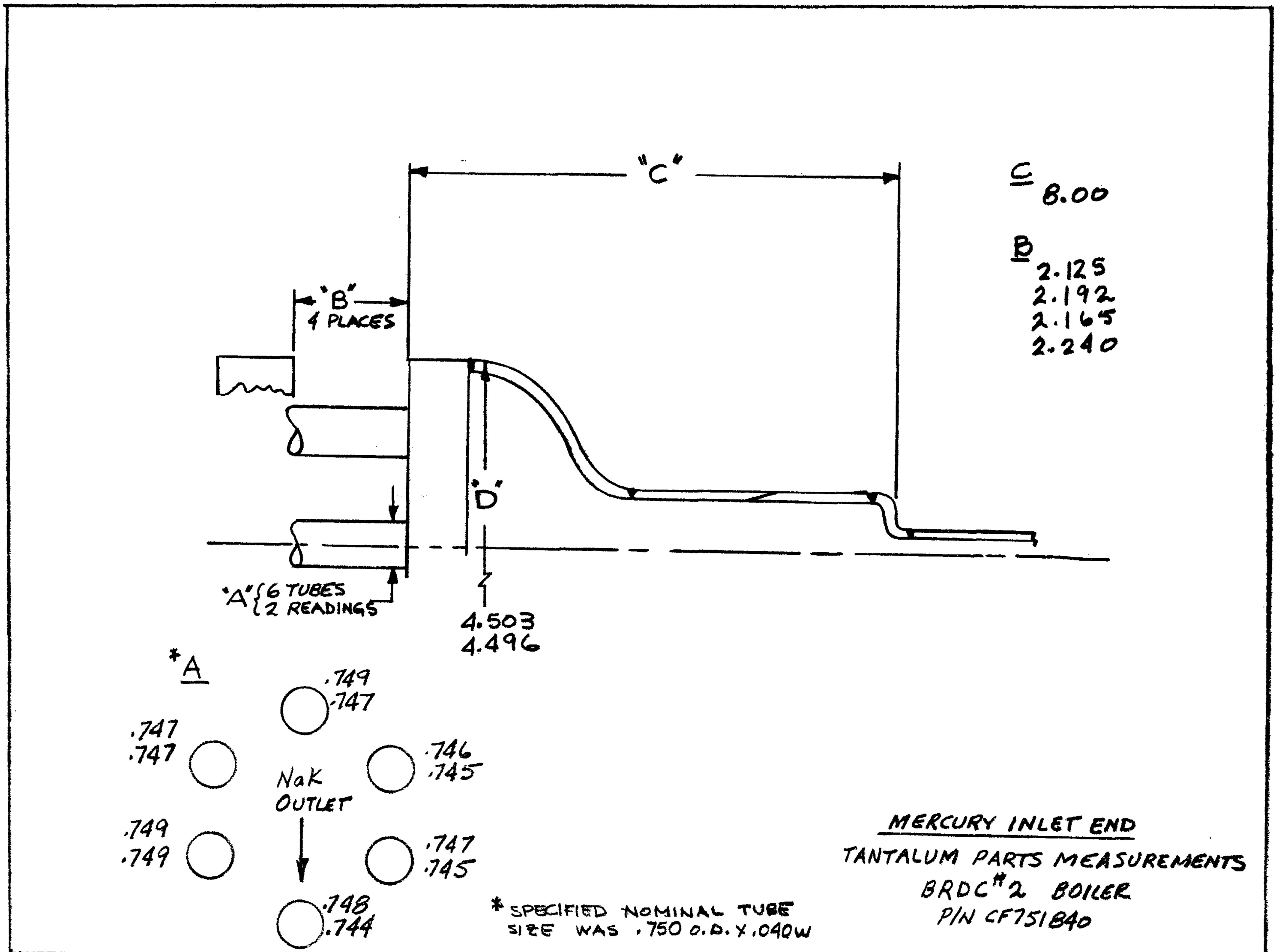
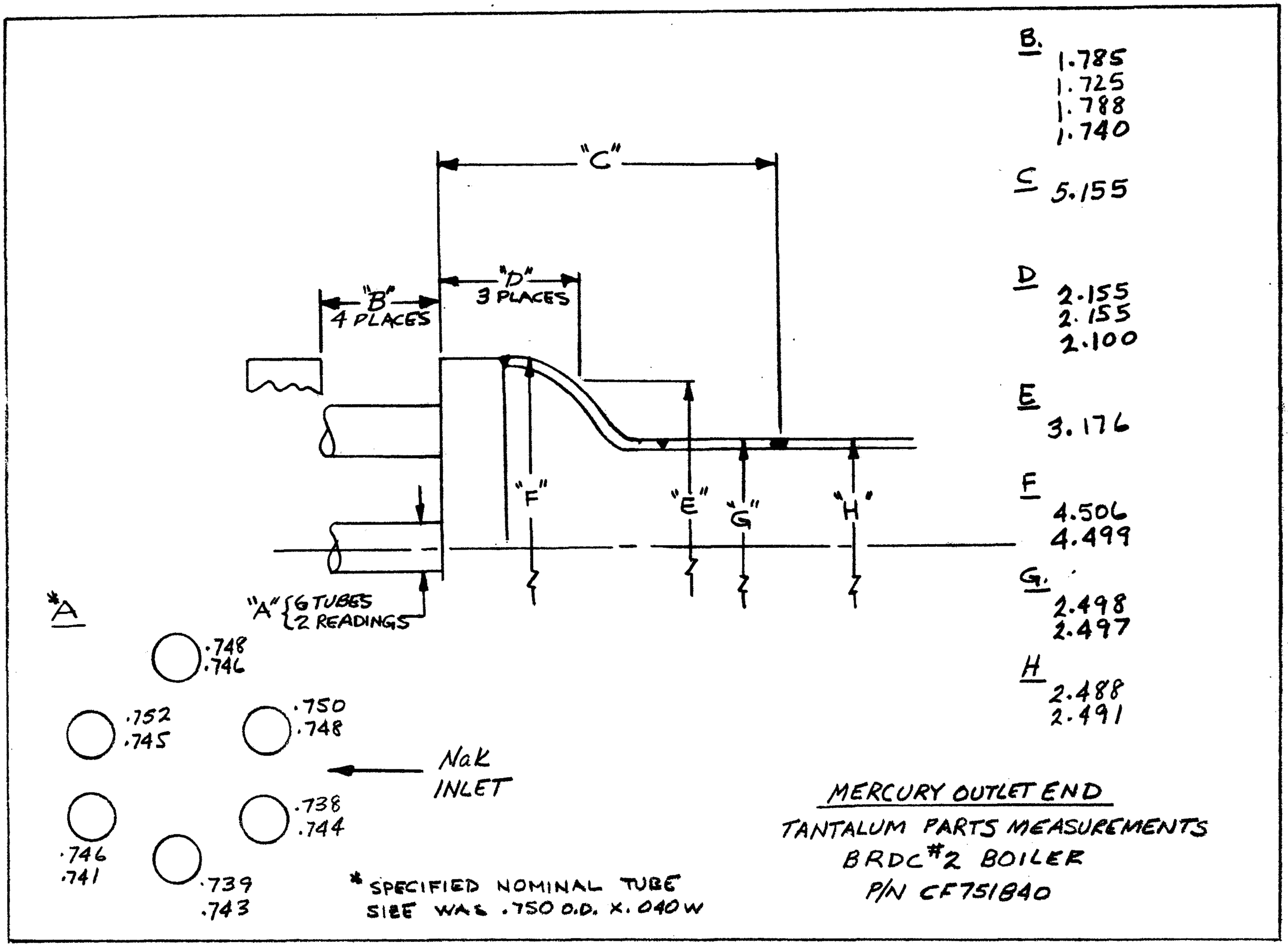
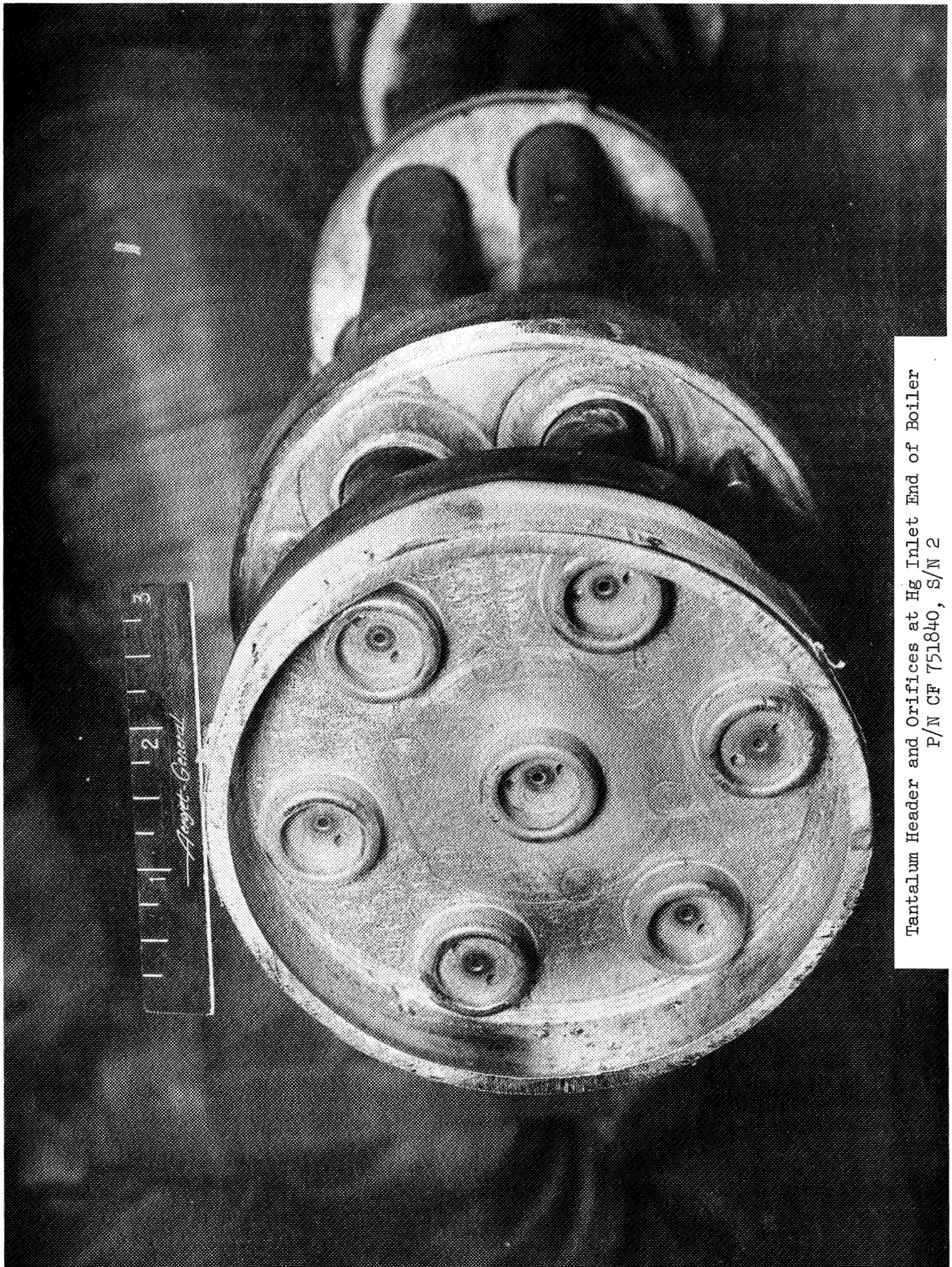

"C"
C 8.00
B 2.125 2.192 2.165 2.240
"B" 4 PLACES
"D"
"A" {6 TUBES 2 READINGS
4.503
4.496
*A
.749 .747
.747 .747
.746 .745
NaK OUTLET
.749 .749
.747 .745
.748 .744
* SPECIFIED NOMINAL TUBE SIZE WAS .750 O.D. X .040W
MERCURY INLET END
TANTALUM PARTS MEASUREMENTS
BRDC #2 BOILER
P/N CF751840

FIGURE 20



"C"
"B"
4 PLACES
"D"
3 PLACES
"F"
"E"
"G"
"H"
"A" {6 TUBES 2 READINGS
*A
.748
.746
.752
.745
.750
.748
.746
.741
.738
.744
.739
.743
NaK INLET
* SPECIFIED NOMINAL TUBE SIZE WAS .750 O.D. X .040 W
B.
1.785
1.725
1.788
1.740
C
5.155
D
2.155
2.155
2.100
E
3.176
F
4.506
4.499
G.
2.498
2.497
H
2.488
2.491
MERCURY OUTLET END
TANTALUM PARTS MEASUREMENTS
BRDC #2 BOILER
P/N CF751840

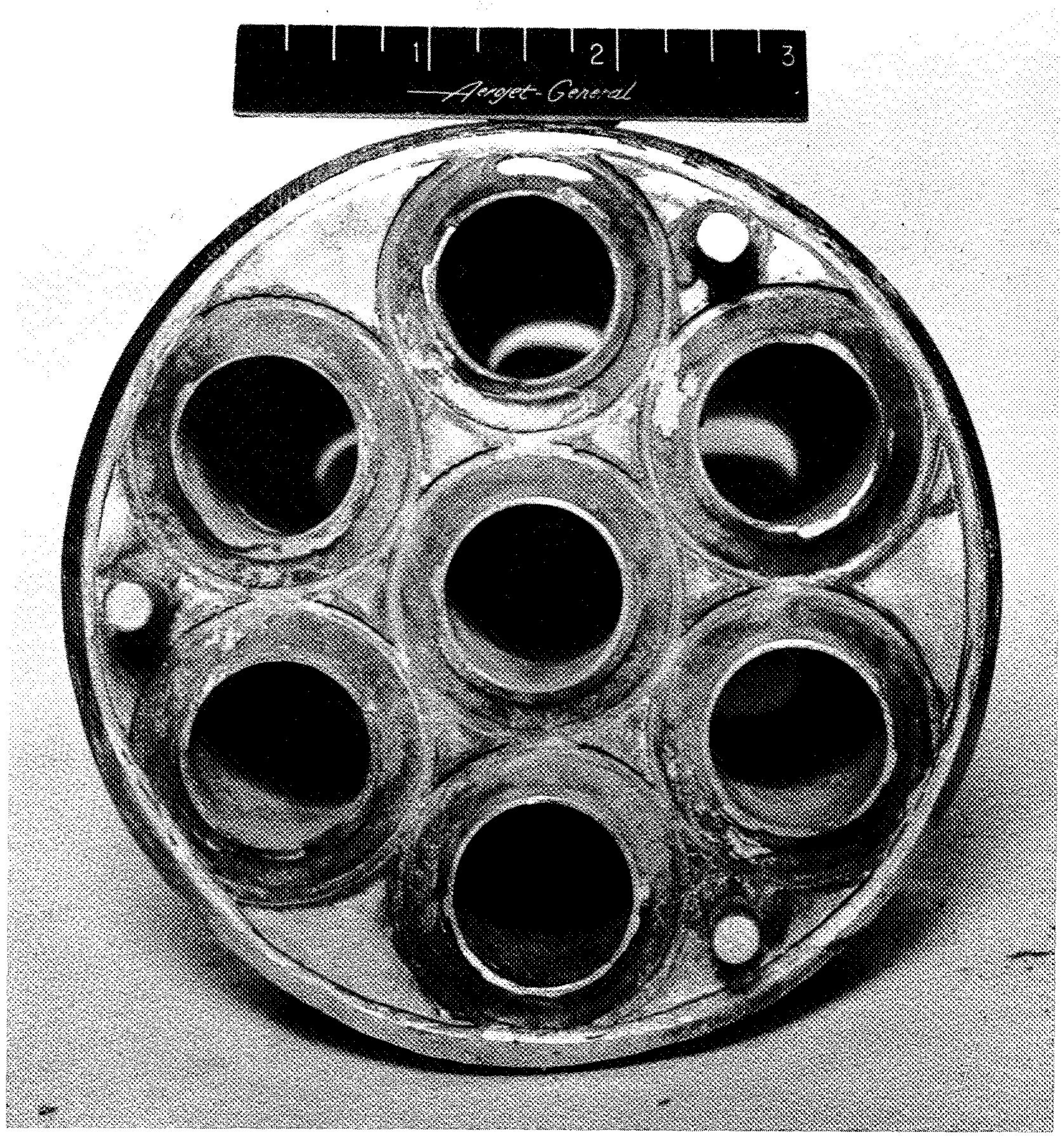
FIGURE 21



Tantalum Header and Orifices at Hg Inlet End of Boiler
P/N CF 751840, S/N 2

1169-096

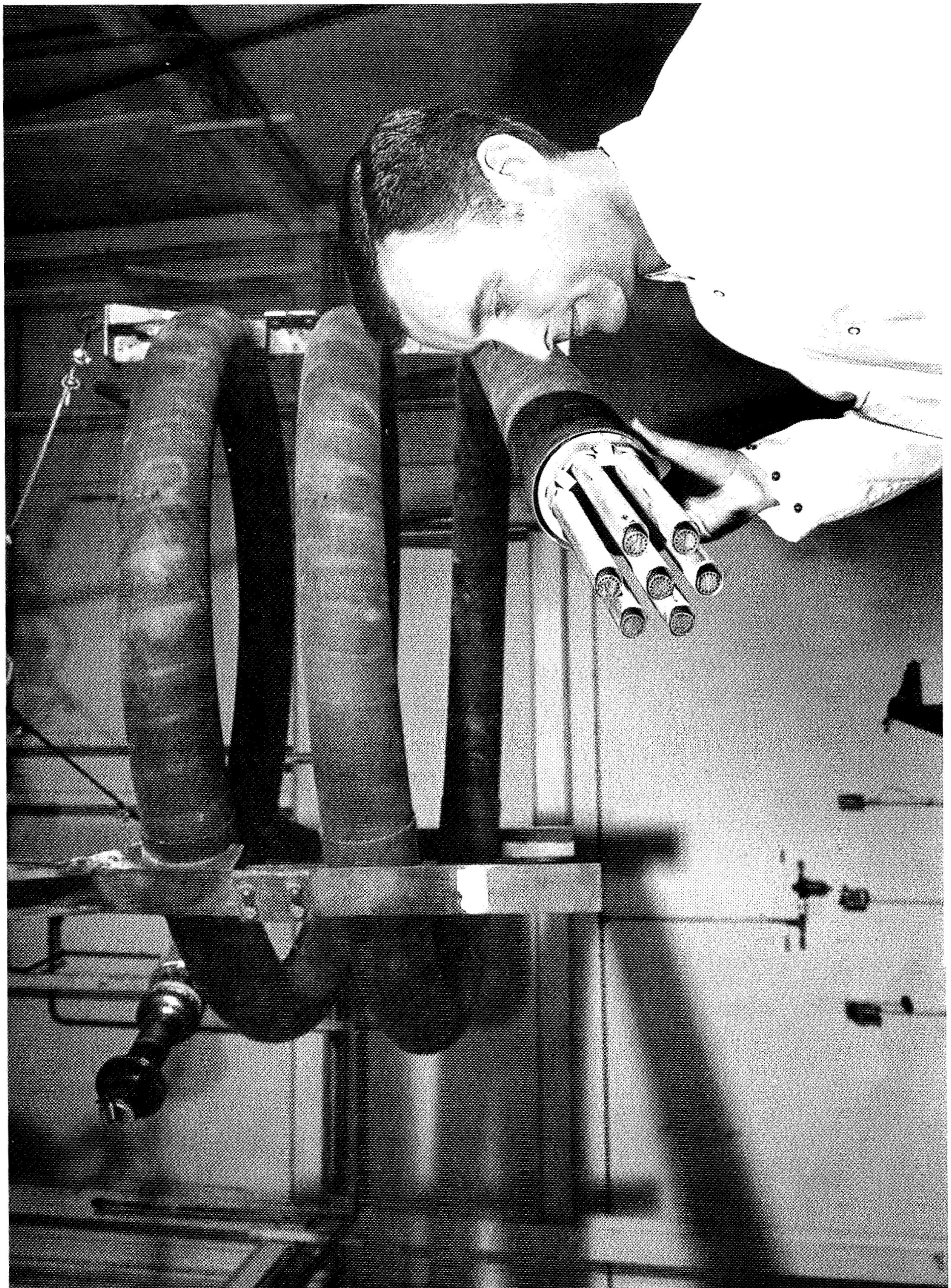
Figure 22



316 SS HEADER-TO-321 SS TUBE WELDS REMOVED FROM BOILER
P/N CF751840-9/2 AFTER 8700 HOURS OF OPERATION

Figure 23

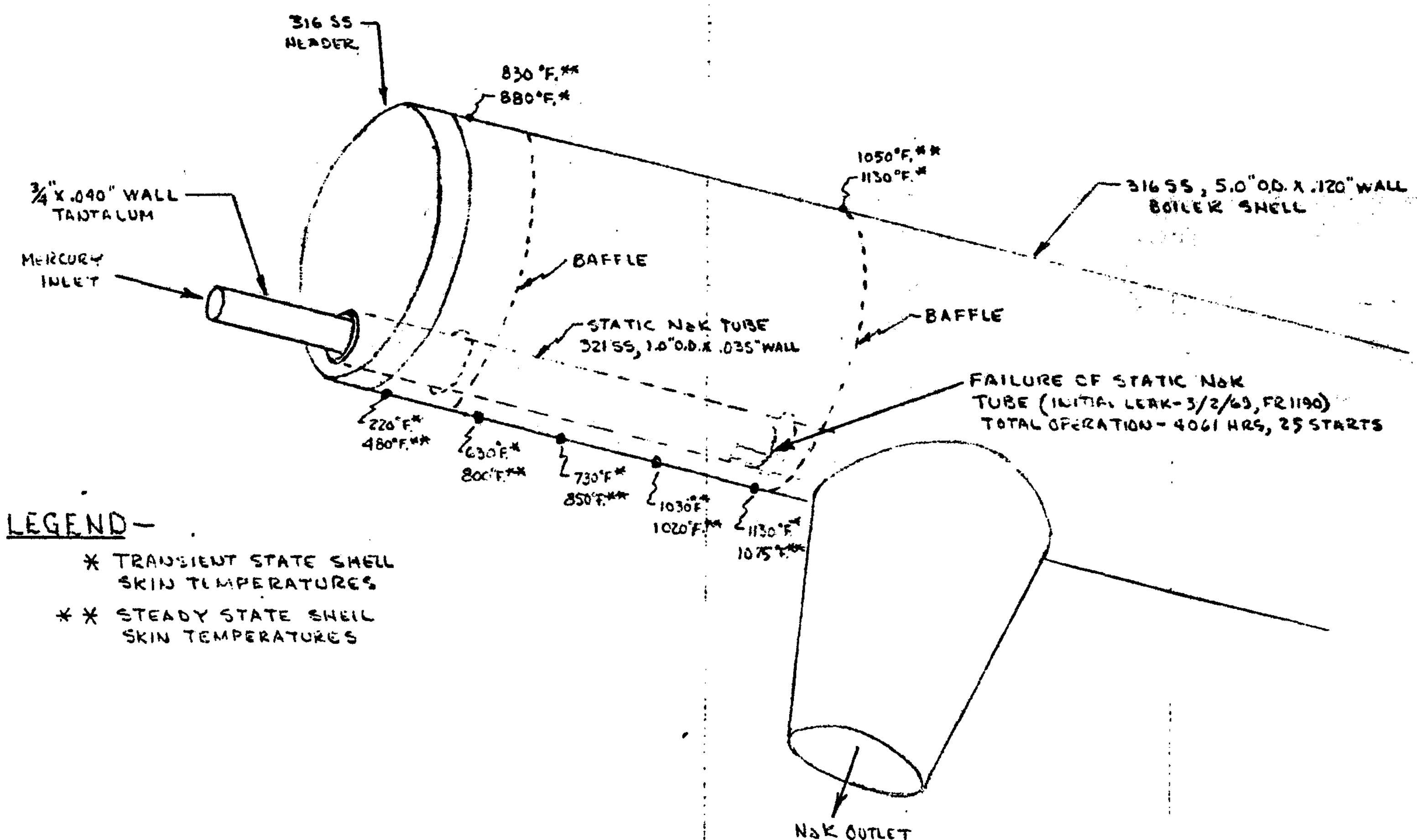
1269-053



State of Boiler P/N CF 751840 S/N 2 After Disassembly

Figure 24

P/N CF 751840 BRDC BOILER 9/2, UNIT #2
TEMPERATURE PROFILE-NaK SHELL
DURING STARTUP & STEADY STATE
OPERATION IN PCS-1 ON 9-4-68



10/9/69 ESC

Figure 25

APPENDIX A

AEROJET-GENERAL CORPORATION

INTEROFFICE MEMORANDUM

23 January 1970
4923:70:0011:HEB:gw

TO: E. S. Chalpin

FROM: H. E. Bleil

SUBJECT: Evaluation of BRDC No. 2 Boiler After 8700 Hours of Operation in PCS-1

COPIES: W. F. Banks, C. E. Hawk, A. H. Kreeger, G. L. Lombard, L. P. Lopez, R. W. Marshall, Jr., H. M. McCall, U. A. Pineda, File

NASA: E. Furman, J. Gentry, R. Miller, P. Stone

REFERENCE:
(a) SNAP-8 Failure Report No. 1067 - 6/6/68

(b) SNAP-8 Failure Report No. 1190 - 3/4/69

(c) SNAP-8 Failure Report No. 1195 - 5/27/69

(d) S. Krikopulo "Crack on the Static NaK Oval Tube" Analysis No. SA-B-240 - 12/2/69

(e) H. Bleil "Thermal Exposure Evaluation of Tantalum/316SS Transition Joints" TM 4923:69-579 - 12/2/69

ENCLOSURE:
(1) Figure 1 - Ta-10w Wire Removed from Turbine Hg Inlet Filter

(2) Figure 2 - Cross Section through Ta/316SS Transition Joint from Boiler Hg Inlet

(3) Figure 3 - Cross Section through Failure Area of 321SS Static NaK Tube

I. SUMMARY, CONCLUSIONS AND RECOMMENDATIONS

Sections of the BRDC No. 2 boiler were evaluated metallographically and by chemical analysis after 8700 hours of operation. The condition of most of the materials in the locations evaluated was excellent. No evidence of oxidation or Hg corrosion/erosion of the tantalum due to system operation was detected. Also, no direct evidence of NaK corrosion existed except at a static NaK tube fracture edge where chromium had been leached from the surface. Mass transfer products, generated in the high temperature areas of the PNL such as gas fired heaters, were present at the boiler NaK outlet to a thickness of 0.0015 inches. At the boiler HG inlet an extremely thin layer of mass transfer deposits was present but none was present at the vapor outlet. In addition, carbon was detected at the Hg inlet on the I.D. of the Ta tubes indicating that organic decomposition contributed to the decreased boiler pressure drop which occurred. The following conclusions resulted from the investigation:

1. No detectable corrosion or erosion of the Ta occurred in 8700 hours of boiler operation.

2. Operation of the boiler in the PCS-1 system did not result in oxidation of the BRDC No. 2 tantalum.

3. As operated in PCS-1, the life of the BRDC No. 2 boiler is not 10,000 hour life limited by either corrosion or erosion. A projected corrosion/erosion life of 40,000 hours does not appear overly optimistic.

4. The decreased boiler pressure drop with increasing operating time is at least partially attributable to organic contamination of the inside surfaces of the Ta Hg containment tubes.

5. The debonding of the hot co-extruded Ta/316SS transition joint at the Hg inlet was typical of the failure mode previously established for this type of joint by laboratory testing.

6. Hydrogen embrittlement combined with residual assembly stresses caused the failure at the Ta-10w swirl wire.

7. The static NaK tube failure was caused by excessive stress, inherent in the boiler design, occurring cyclically during system startup and shutdown.

8. Localized wall thinning of the Ta tube under the failed static NaK tube was associated with oxidation and/or erosion as a result of the crack in the static NaK tube.

9. The zirconium foil served its intended function of purifying the static NaK by absorbing oxygen, nitrogen and carbon.

10. Oxidation of the O.D. of the Ta Hg inlet dome most likely occurred during a boiler modification and repair welding operation. Subsequent boiler operation confirmed that NaK removes oxygen from tantalum.

The following recommendations are made:

1. Design modifications should be made to reduce the stress level in the static NaK tube at the Hg inlet and in other areas if stress analysis indicates failure is probable.

2. Optimization of the fabrication techniques for the production of reliable Ta/316SS transition joints should be continued, and the resultant best products tested to establish their acceptability for SNAP-8 boiler use.

3. PCS-1 operational modifications and improvements should be made to eliminate organic contamination of the Hg loop.

4. The conditions which produced the Ta tube wall thinning under the crack in the 321SS static NaK containment tube should be simulated to determine if this is a potential failure mode, and to establish the mechanism causing the thinning.

5. The chemical cleaning procedure, AGC 10319/8, should not be used for boilers containing Ta-10w turbulator wire. An alternate material, not subject to hydrogen embrittlement when so cleaned, should be considered or a different cleaning procedure should be developed for removing heat transfer inhibiting surface films from partially deconditioned Ta boiler tubes.

II. INTRODUCTION

The SNAP-8 bare refractory double containment mercury boiler No. 2 was first operated in PCS-1 in March 1968. After 1313 hours of operation the boiler was repaired following an Hg outlet bellows failure, (Reference a). After approximately 4000 hours of operation a PNL to static Nak leak was detected, (Reference b), but operation was continued since the leak did not affect the overall performance of the boiler. After 6138 hours of operation a NaK to air leak occurred in the 316SS boiler shell at the NaK outlet, (Reference c). This was repaired by welding a doubler on the shell over the leaking area and operation was continued. In September 1969, after a total of 8700 hours of Hg operation which included a total of 28 startup-shutdown cycles, the boiler was removed from PCS-1, and portions of it were removed for evaluation. During its operational life this boiler had produced Hg vapor for turbine operation in an excellent manner. Although the performance of the boiler did degrade during its life as indicated by a decreased pressure drop between the Hg inlet and outlet, its overall thermal performance was unchanged.

III. Ta DOME AT Hg INLET

Metallographic examination of the Ta dome section joining the Ta/316 transition joint to the Hg tube header was performed. A Ta weld had been made in this area in repairing the boiler after the outlet bellows failure after 1313 hours of operation, (Reference a). Although the weld was excellent, the parent metal on both sides of the weld showed indications of having been oxidized. This manifested itself as intergranular and preferential crystallographic plane penetration of the Ta on the O.D. of the dome. It is probable that this condition occurred during the boiler repair welding as a result of the Ta being heated in the presence of oxygen. An indicated by polarized light examination, it also appeared that the oxide which had formed was removed by the static NaK during subsequent system operation. Hardness measurements also indicated that oxygen contamination of the Ta in this area was not present at the time of examination. This is the only area os the boiler Ta which was examined which gave any indication of having been oxidized. It also confirms, as was anticipated, that at elevated temperature, NaK will remove oxygen from tantalum.

IV. Ta ORIFICE AND TUBE AT Hg INLET HEADER

A longitudinal section through one of the Hg inlet orifices was examined metallographically. No indications of corrosion, erosion, or mass transfer product deposition were detected. On this same tube, where it exits from the header on the upstream side, smooth slight depressions were present to a depth of 0.0004 to 0.0015 inches into the tube. This indicates that tube bending stresses at the header during boiler operation did not deleteriously affect the Ta tubes. The Ta tube/header/orifice weld was excellent in quality. As anticipated the weld had a larger grain size than the parent metal but no indications of oxidation or corrosion were detected metallographically or by microhardness determinations.

V. Ta TUBES AT Hg INLET

Approximately one foot of the Ta tubes from the Hg inlet end had been cut from the boiler for examination. The tubes were sectioned longitudinally and the I.D.'s were examined visually and metallographically. No evidence of Hg corrosion/erosion of the Ta tubes or plugs was detected either visually or metallographically. Each tube was silvery-colored with a slight yellow tinge on the Hg side at the Hg inlet. This graded into a bluish-black discoloration just before or just after reaching the fluted area of the plug. The dark discoloration was not present in the areas where the top of the flutes had been in contact with the tubes. The darker discoloration appeared to be an extremely thin film which could not be detected metallographically. By careful scraping, some of the film but including some of the underlying Ta, was collected from one tube and analyzed. The results of the normalized analysis indicated the film was approximately 93% Fe, 3% Ni, and 3% C. The Fe and Ni were undoubtedly Hg mass transfer products. It is presumed that the carbon is the result of decomposition of organics which entered the Hg system. Mix 4P3E, octoil, and duoseal oil have frequently been detected in small quantities in various portions of the Hg system. An infrared spectrographic analysis of solvent washings from the I.D. of one of the tubes indicated the presence of duoseal oil. Although the pattern of deposition in the Ta tubes through the remainder of the boiler is not known, the presence of carbon at the Hg inlet indicates that the decreasing pressure drop which was noted across the boiler may have been contributed to strongly by organic contamination. No visual, metallographic or hardness measurement indications of oxygen contamination of the Ta tubes were detected. This indicates that the vacuum and inert atmospheres maintained in the Hg system during heat-up prior to Hg injection and during cool-down after stoppage of Hg flow were adequate to prevent deleterious contamination of the Ta. This also indicates that oxide formation on the I.D. of the Ta tubes was not the cause of the decreasing boiler pressure drop.

VI. Ta-10W SWIRL WIRE

A two-inch long section of wire was found in the turbine Hg inlet filter after it was removed from PCS-1. Chemical analysis revealed the wire was Ta-10w, the material used for the swirl wire in the boiler. Boroscope examination revealed that a section of this wire was missing from the center Hg tube of the boiler at the outlet end. Metallographic examination of the wire removed from the filter revealed it contained numerous cracks, (Figure 1), however, the wire could be free bent 180° without further cracking. Sections of Ta-10w wire which had been used for coiling experiments and new wire procured for the fabrication of another boiler could also be bent 180° without fracturing. Metallographic examination did not reveal the presence of cracks in these two wires. All three wires were in the cold worked condition and each had the same hardness, Rockwell C 38. Cleaning per AGC 10319/8 of the two sections of wire which had not been in the boiler resulted in their becoming embrittled. In bending these sections of wire, fracture occurred with essentially no permanent set. It was presumed that hydrogen embrittlement of the wires occurred during cleaning. Separate sections of wire were electrically coupled to 316SS and to platinum during exposure to 2-1/4 pounds KOH per gallon of water (that solution in the cleaning operation most likely to cause embrittlement) at 210-220°F for 1-1/2 hours to determine if these materials would provide anodic protection and prevent the embrittlement. Both wires were embrittled after this treatment indicating that anodic protection was not effective. Sections of the embrittled wire were exposed at elevated temperature in vacuum to outgas the hydrogen. Ductility was completely restored, as indicated by bend testing, after exposure at 1000°F for 16 hours or at 1500°F for 2-1/2 hours, confirming that hydrogen had produced the embrittlement. During the life of the boiler, the swirl wire had been exposed to hydrogen several times. During the initial hot outgassing of the boiler, hydrogen was evolved presumably from the tantalum in the boiler. After the bellows failure, the Hg side of the boiler was chemically cleaned, as described above, which would cause absorption of hydrogen not only by the wire but by the Ta tubes. This hydrogen was subsequently removed by hot outgassing prior to Hg injection and during the subsequent system operation. After the failure of a PNL line at the gas fired heater outlet and the concomitant replacement of a Hg PMA, hydrogen from an undetermined source was evolved in the Hg loop. Also, organics have been detected in the Hg system; decomposition of these materials produces hydrogen.

It was concluded that hydrogen embrittlement of the Ta-10w swirl wire in the boiler had occurred at least one time during the operation of the boiler in PCS-1. This embrittlement in conjunction with residual stresses in the wires from coiling and from inserting them in the boiler tubes had caused the failure of the wire. Subsequent system heatup and operation had outgassed the hydrogen from the wires restoring their ductility. In addition to the piece of wire which had completely broken loose from the boiler, other wire cracking may have occurred which could have resulted in additional loss of wire from the boiler during continued operation. This potential condition in conjunction with other deleterious conditions in the boiler made it undesirable to continue operating with this component.

VII. Ta/316SS TRANSITION JOINT AT Hg INLET

The hot co-extruded Ta/316SS transition joint at the boiler Hg inlet was partially debonded. Hand held transducer, ultrasonic inspection revealed that the 316SS had debonded on the O.D. for a longitudinal distance of approximately 1/8 inch around the entire circumference of the joint. In addition, one area indicated debonding approximately 1-1/2 inches around the circumference for a longitudinal distance of approximately 1/2 inch. Since previous laboratory exposures of this type of joint had indicated that debonding would continue to failure of the joint and subsequent leakage, (Reference e), the joint was destructively sectioned for metallographic examination. In the area where ultrasonic inspection had indicated debonding to a depth of 1/2 inch, the metallographic examination revealed debonding for a distance of 0.323 inches on the O.D., Figure 2. In addition, on the I.D., the tantalum had debonded from the 316SS for a distance of 0.012 inch in the plane examined. Both the 316SS and the Ta were in the cold worked condition and at their interface a third phase was present. These conditions are typical of those found in the samples, produced by the same technique, which were previously investigated. A program is presently in progress under the control of NASA LeRC to improve and optimize the fabrication techniques and reliability of hot co-extruded Ta/316SS joints. No evidence of Hg corrosion or mass transfer product deposition were noted on either the 316SS or the Ta of the transition joint.

VIII. 321SS STATIC NaK TUBE FAILURE

Another undesirable condition was cracking of the lowest 321SS static NaK containment tube which failed in the transition from round to oval cross section at the Hg inlet. This cracking was a cause of leakage between the static NaK and the PNL, but sectioning and post operational testing of the boiler was not continued to confirm that this was the only leakage path. Stress analysis indicated that this tube had been subjected to a stress of 44 KSI and that failure could be attributed to cyclic thermal stressing which occurred during the heating and cooling of the boiler for system startups and shutdowns, (Reference d). A factor which caused additional stress in the failed tube but which was not included as part of the stress analysis was the longitudinal rubbing and galling of the bottom of the tantalum tube with the 321SS tube surrounding it. This was detected both visually and metallographically.

Visual examination of the failed tube revealed that although the cracking had undoubtedly occurred under tensile loading, stress reversal had forced the fracture surfaces together under compressive loading so that the edges of the crack were cold worked and extruded both outward and inward slightly. Metallographic examination revealed that the 321SS had a normal structure except at the fracture surfaces where compressive yielding had occurred, (Figure 3). The general structure was annealed, equiaxed grains. Less than one percent sigma phase was present. This structure was identical with the structure of the top 321SS tube which had not failed. Each tube in the boiler at this location is subject to different stress levels because of the thermal gradients in the area. At the compressively yielded fracture surfaces of the failed tube a different metallographic structure existed, although X-ray diffraction indicated the presence of only austenite. The cold worked area was slightly lighter in color in the unetched condition and appeared as "salt and pepper" in the etched condition. Annealing the fractured material at 1900°F did not change this structure. A step scan microprobe analysis for Fe, Cr and Ni across the crack surface structure into the unaffected parent metal revealed that at the crack surface the analysis was Fe - 79%, Cr - 8.7%, and that the analysis changed approximately linearly across the abnormal structure to Fe - 69%, Cr - 18%, in the unaffected material. The Ni concentration was constant across this area. The zone of abnormal structure was approximately 0.00157 inches thick in the area analyzed. The cause of the chemical gradient across the cold worked zone is not completely understood. Since the tubing was exposed only to NaK at elevated temperature, it is presumed that this eutectic alloy with low oxygen content caused the preferential leaching of chromium from the fracture surface. Possibly the higher energy state due to the cold working of the fracture edges contributed to leaching at the fracture edges than was apparent in the remainder of the tubing. In addition, with changes in PNL or static NaK pressure, a flow of NaK through the crack up to an estimated velocity of 17 ft/sec occurred. The velocity of the PNL NaK over the O.D. of this tube is 4-1/2 ft/sec. The higher flow rate through the crack may also have contributed to preferential leaching in this area.

IX. Ta TUBE WALL THINNING UNDER STATIC NaK TUBE CRACK

Although the failed section of the 321SS tube could have been replaced, the Ta Hg containment tube immediately under the crack had suffered localized wall thinning. Metallographic examination revealed that as much as 0.0185 inch of the 0.040 inch thick wall had been removed on the O.D. of the Ta tube immediately under the crack in the static NaK containment tube. Again the reason for this localized wall thinning is not clear. Metallographically the thinning was smooth rather than rough, no differences in hardness or structure were visible in the thinned area as opposed to unthinned areas. No evidence of subsurface oxidation was present in any location on the Ta tubes. It is thought possible that short term, low temperature surface oxidation of the Ta may have occurred due to oxygen entering the static NaK system through the cracked 321SS tube during the time between the failure and repair of the boiler shell. The oxygen would have to be supplied from the PNL where the NaK/air leak occurred. The possibility of this occurring was minimized by keeping both the PNL and static NaK system under argon pressure after the failure occurred until both systems were evacuated to be refilled with NaK. The argon used did contain some oxygen. Localized formation of sodium tantalate on the Ta tube under the crack and subsequent solution or erosion by the NaK flowing through the crack of this material or of the Ta itself may have produced the wall thinning.

X. ZIRCONIUM FOIL IN STATIC NaK SYSTEM

The corrugated Zr foil getters which were wrapped around the inlet and outlet Ta/316SS transition joints in the static NaK system, were removed after the boiler had been decontaminated of NaK. The foil from the NaK outlet was discolored dark grey and was friable at all locations. The foil from the NaK inlet was discolored grey and was friable in the outer layers but was clean silvery metallic colored on the I.D. side of the inner wraps away from the edges. These clean areas could be bent without breakage. Carbonates were present on the foil from both areas. Presumably these formed due to absorption of carbon dioxide from the air by the residual NaK after the boiler had been cut from the system. The variability in color and friability of the foil from the NaK inlet area indicates a variable degree of contamination of the Zr by C, H, N, and/or O, within the wraps of the foil and produced presumably by the shielding of the inner wraps by the outer wraps. Chemical analysis of the foils for C, H, N, and O tended to confirm this variability. From the analysis results obtained, only qualitative conclusions can be drawn. The carbon, oxygen, and hydrogen content of both foils was higher than in the original foil. The hydrogen increase may have, in part at least, occurred during decontamination. The foil at the NaK inlet end of the boiler showed an increase in nitrogen content, but the foil at the outlet did not and may actually have decreased in nitrogen content. The increase in nitrogen in the inlet foil presumably is due to absorption from the NaK of nitrogen which was dissolved in the NaK at the argon/NaK interface in the expansion tank. The closer proximity of the inlet foil to the expansion tank and the static condition of the NaK would make the transfer of the nitrogen impurity in the argon essentially a diffusion process. As such, it would be expected to be gettered preferentially by the inlet foil. It appears that the Zr foil served its function of purifying the static NaK during boiler operation.

The following photographs were taken during the course of this evaluation and are available in Department 4923 Files for examination.

A 3599, 3602, 3603
A 3616 through 3621
A 3623 through 3625
A 3628 through 3630
A 3646 through 3651
A 3661 through 3674
A 3696 through 3714
A 3716
A 3719 through A 3721
A 3739, 3740, 3743, 3749

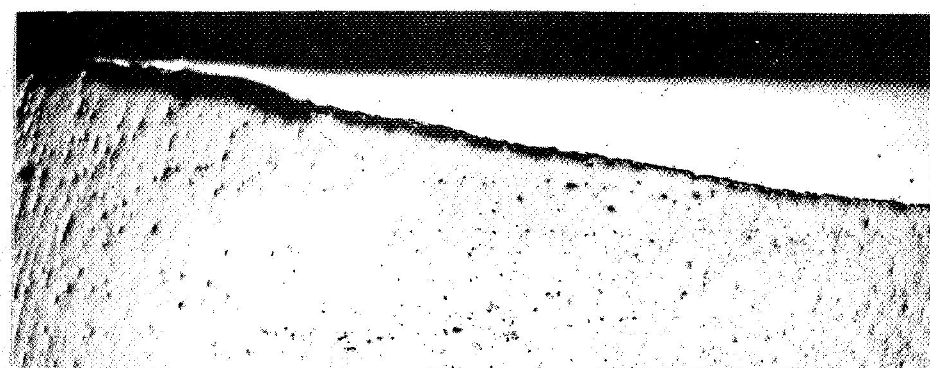
H E Bleil
H. E. Bleil
Met. Eng'r and Mech. Test
Engineering Analysis
Power Systems Department

Approved:

HDerow
H. Derow, Supervisor
Met. Eng'r and Mech. Test
Engineering Analysis
Power Systems Department

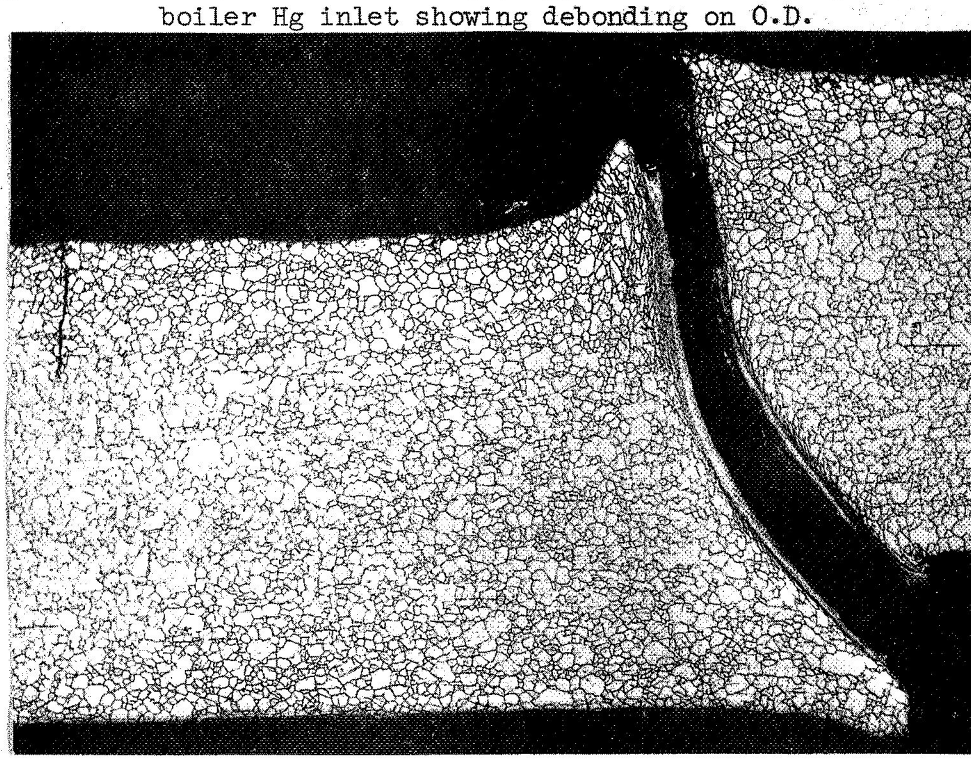
20X A 3603

Figure 1 - Ta-10w wire removed from turbine Hg inlet filter.



40X A 3740

Figure 2 - Cross section through Ta/316SS transition joint from boiler Hg inlet showing debonding on O.D.



75X, Ammonium persulfate electrolytic etch A 3661

Figure 3 - Cross section through failure area of 321SS static NaK tube.

APPENDIX B

Dept. 4927

ANALYSIS NO. SA-B-340

DATE 12-2-69

SUMMARY OF ANALYSIS

Project SNAP-8	Component LeRC Boiler	Distribution:
Part Static NaK Oval Tube	Drawing No. Sketch	E. Chalpin
Subject Crack on the Static NaK Oval Tube		G. Lombard
Reference(s)		E. Furman
Engineer S. Krikopulo	Approved W. Wolff	File: SS 1020-03

OBJECTIVE: What caused the crack on the static NaK oval tube.

ASSUMPTIONS:

Temperature chart and NaK pressure are given.
Assumed parabolic radial gradient.
Assumed surface contact between mercury tube and NaK tube.

REFERENCES (Analysis Methods):

Conventional and Finite Element Computer Program.

RESULTS AND CONCLUSIONS:

Total max. stress in the oval tube - 44,500 PSI.
Fatigue life based on the above stress: 100 cycles.
Cause of crack: combined loads rather than a single one. These loads are due to:

1. Axial and radial thremal gradient.
2. Circumferential thermal gradient in the outer shell.
3. Differential pressure of 10 PSI.

RECOMMENDATIONS AND COMMENTS: